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A Machine for Laboratory Washing Tests[1-2]

Description of Laboratory Washing Machines—Control of Mechanical Action—Other Uses for the Machine —Proposed General Specification for a Standard Laboratory Washing Machine—Proposed General Specification for a Standard Laboratory Washing Procedure

By WM. D. APPEL[3], WM. C. SMITH[4] and HUGH CHRISTISON[5]

ABSTRACT

Co-operative work by the American Association of Textile Chemists and Colorists and the Bureau of Standards has resulted in a practical machine for testing the fastness to washing of dyed textiles. With it one man may test up to twenty samples simultaneously and in a brief run obtain results similar to those obtained in commercial laundry practice. It is also suitable for testing detergents, for dry-cleaning tests and for laboratory dyeing. The machine is described and general specifications for it and for a standard washing procedure are given. The machine is being manufactured and may be obtained through the Association.

Introduction

ALTHOUGH a number of operators working independently in different laboratories are able to duplicate one another's results in laboratory washing tests carried out in beakers[6], in the usual way, it is obvious that this method is open to serious criticism. It requires constant attention. Changes in concentration of the wash solution through evaporation are difficult to control. The tests are deficient in "suds," according to the laundryman. An adequate substitute for the mechanical action that usually accompanies any sort of laundering is lacking. Mechanical action may not be an important factor in testing certain fabrics and some dyes, but it is of great importance in testing cotton goods and such dye types as the vats, dyes, pigment colors, and coupled "ice" colors.

The laboratory washing machine described below has been developed to take the place of the beaker test in the standard methods of the American Association of Textile Chemists and Colorists[7] for testing fastness to washing of colored textiles. It is free from the faults mentioned and makes possible strictly reproducible results. It has been found to give essentially the same results as commercial laundering in the removal of soil, and for this reason is under consideration by the Detergents Committee of the American Oil Chemists' Society for standard tests of detergents. The machine is simple in construction, requires little attention while it is running, and one man can turn out a large amount of work with it. A very practical form of the machine is now available on the market.

Several modifications of the machine are described and illustrated in this paper and various uses indicated. Since it seems to be well suited to the standardization of laboratory washing tests, a proposed general specification for the machine and for a standard washing procedure are given.

Description of Laboratory Washing Machines

The first machine to be built and the simplest in construction is shown in Fig. 1. It has been in successful operation at the Arlington Mills, Lawrence, Mass., for some years. Half-pint glass-topped preserve jars hold the samples and their wash solutions. They are arranged radially in a galvanized iron drum which is mounted on a shaft and rotated by means of a small electric motor. The wash solutions are heated to the desired temperature before placing in the jars, and the temperature is maintained by heating the drum from below with a gas burner. With a little experience, the temperature can be kept constant within a few degrees. When the drum is rotated, the sample and solution falls from one end of the

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[1] Presented before the Division of Dye Chemistry at the seventy-sixth meeting of the American Chemical Society, Swampscott, Mass., September 10-14, 1928.

[2] Publication approved by the Director of the National Bureau of Standards, Department of Commerce, Washington, D. C.

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[5] Chief Chemist, Arlington Mills; Chairman Sub-Committee on Fastness to Washing of Dyed Wool, Research Committee, A. A. T. C. C.

[6] American Dyestuff Reporter, **15**, 871 (1926).

[7] See the Year Book, American Association of Textile Chemists and Colorists.

jar to the other and good suds are produced. Thus the conditions in a commercial laundering machine or a home electric washer are simulated.

Another type of machine, built at the Bureau of Standards, is shown in Fig. 2. It differs from the Arlington

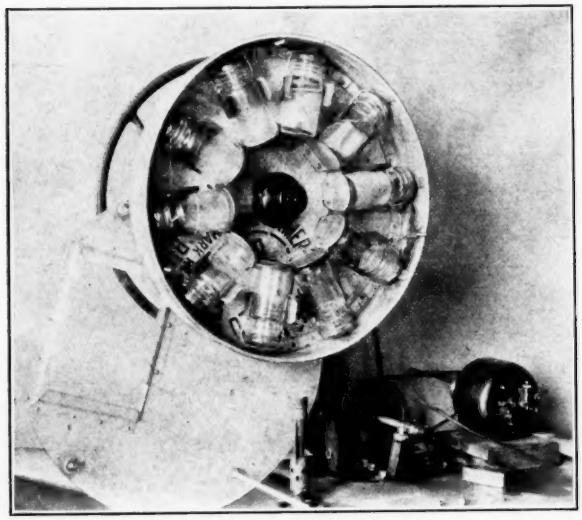


FIG. 1—*Arlington Mills Machine*

Mills machine in using one-pint instead of half-pint jars, in their arrangement on either side of a horizontal shaft, and in the use of a water bath instead of an air bath to control the temperature. This machine is driven directly by the motor through a worm gear. The temperature of the water bath does not fall rapidly, and even at 160° F. the use of a burner below the tank is not necessary. Hot water added at intervals will keep the temperature within one or two degrees of that desired. The removal of the jars, which are completely submerged when this machine is in operation, is difficult unless the water is drained off.

A machine which combines the advantage of the accessibility of the jars in the Arlington Mills machine with that of the better temperature control of the Bureau of Standards machine was built at the Pease Laboratories, New York, under the direction of Dr. L. C. Himebaugh, and has been in successful operation for nearly a year. It is shown in Fig. 3. A somewhat improved model has just been placed on the market for the American Association of Textile Chemists and Colorists. It is shown in Figs. 4 and 5. The tank serves as a water bath in which rotates the shaft carrying four rows of jars, five in a row, arranged radially. The speed of rotation is 42 r.p.m. A clutch is provided so that the shaft may be stopped without stopping the motor. One-pint glass-topped preserve jars are used. They have a height of about 6 inches and diameter of 3¼ inches. The neck is but slightly constricted. Although the jars may be completely covered with water when this machine is in operation a depth of 11 inches gives satisfactory temperature control. The jars are thus out of the water and easily accessible when they turn to the upper position. A gas burner is provided below the tank to regulate the temperature of the water in it. Without external heating and under normal working conditions a drop in temperature of only about 5.0° from 160° F. was noted in 20 minutes. A large drain pipe is placed in the bottom of the machine and a thermometer well is provided. It is convenient to use a soap solution instead of pure water in the tank to avoid splashing.

CONTROL OF MECHANICAL ACTION

When samples of artificially soiled cotton cloth[8] are washed alone in machines of this type, they are not as well cleansed as in commercial laundering. In order to increase the mechanical action small cubes of heavy rubber were introduced into the jars with the samples. Later, balls of rubber and of metal were tried. Samples of the soiled cloth washed with rubber balls 1 cm. in diameter and a specific gravity of 3, were cleansed to the same extent as in the corresponding commercial laundry treatment. It is thought that by using the rubber balls the pounding and rubbing together of the load in a commercial machine is satisfactorily duplicated in the laboratory.

[8] Guernsey and Howells, J. Oil and Fat Industries, **4**, 390 (1927).

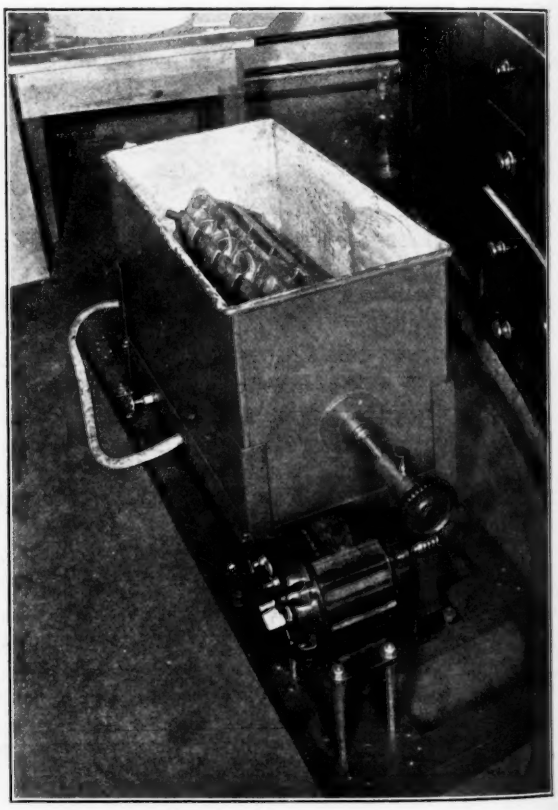


FIG. 2—*Bureau of Standards Machine*

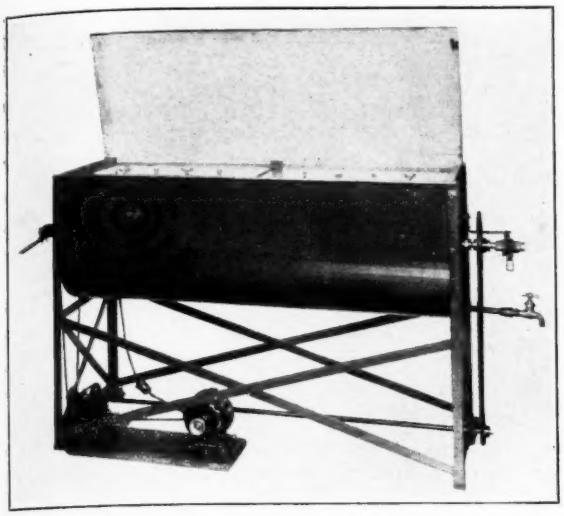


FIG. 3—*Pease Laboratory Machine*

Similar action in a domestic washing machine or in hand washing on a scrubbing board may be equally well represented in a standard washing test.

If metal balls are used instead of rubber balls, still greater mechanical action is obtained. Thus it is possible to have a washing test which is accelerated with respect to mechanical action. The comparative action of several types of balls is illustrated in Fig. 6. Heavy balls produce a greater effect than light ones. Eight or ten balls to the jar produce practically as great an effect as a large number. Three-eighths-inch steel balls have a greater action than an equal number or equal weight of smaller ones. Breakage of jars with steel balls of larger size than ⅜ inch prevents their use unless metal containers are provided. Lead balls are not practicable, because they soil the cloth. Where very great mechanical action is desired—as, for example, in a laboratory fulling test or in dyeing leather—it may be desirable to use large metal balls and metal containers.

The results of experiments with the machine lead to the recommendation that no balls be placed in the jar when a test that is mild with respect to mechanical action is desired, as in washing silk. For tests that are to duplicate the effect of commercial laundering with reference to the removal of dirt or of color, 1-cm. rubber balls, specific gravity 3, are recommended. When an accelerated washing test is desired, ⅜-inch steel or Monel metal balls may be used. For maximum mechanical action metal containers and ⅜ or ½ inch metal balls should be satisfactory.

The machine is obviously suitable for testing detergents as well as for testing the color fastness of fabrics. Laboratory dry-cleaning tests have been satisfactorily carried out in a machine of this kind. It has been common practice to dye leather in the laboratory in jars held in some sort of rotating or oscillating machine. Leather dyeing tests can be standardized by the general adoption of a machine such as this one for the tests. It is believed that the use of balls in the jars in laboratory leather dyeing will materially improve the results by simulating the pounding action of the load in large-scale dyeing.

OTHER USES FOR THE MACHINE

The use of the machine for laboratory fulling tests is under consideration. One important use is in making laboratory dyeings of vat and sulphur dyes. By nearly filling the jars with the dye bath and adjusting the hydrosulphite (or sulphide) used, the scum of oxidized dye in ordinary beaker dyeing can be avoided and no further additions of reducing agents are necessary. Other laboratory dyeing tests, particularly those made at temperatures below boiling, may be made in the machine. The uniformity of agitation and temperature would be greater than in beaker tests with hand turning of the material, and the operator would be free for other work. It might prove desirable to provide special jars with hooks in the top and bottom for the support of skeins during dyeing.

FIG. 4—*Association Model, Side View, Capacity Twenty Jars*

Proposed General Specification for a Standard Laboratory Washing Machine

The requirements of laboratories which test fastness to washing differ widely, and it seems immaterial how the details of construction of the standard washing machine are worked out. The following general specification is suggested as adequately covering a machine for standard washing tests for determining the fastness of colored textiles.

> The standard washing machine shall consist of jars mounted on a shaft which can be rotated at a constant speed of 40 to 45 r.p.m. One-pint glass-topped preserve jars, 6 inches in height and 3¼ inches in diameter, shall be used. They shall be arranged radially about the shaft, the base of the jar being 2 inches from the center of the shaft. Provision shall be made for maintaining the temperature of the wash solution to within ±2° F. of that specified for a given test.

Proposed General Specification for a Standard Laboratory Washing Procedure

The composition of the washing solution, its temperature, the time of washing, the sequence of washing, rinsing, or other operations will vary with the type of test, but certain procedures common to all types can be standardized to advantage. The following general specification is proposed for standard laboratory washing tests made in the standard machine.

Unless otherwise specified, the volume of the liquid placed in the jars for washing the sample shall be 100 c.c., and for rinsing 200 c.c.

Unless otherwise specified, the sample to be washed shall be 10 square inches in area. A piece 2½ x 4 inches in size is convenient. A piece of white cloth of the same dimensions free from sizing shall be sewed to it near one end to be used in judging staining. The particular kind of white material shall be specified in reporting staining.

Fig. 5—*Association Model, Top View*

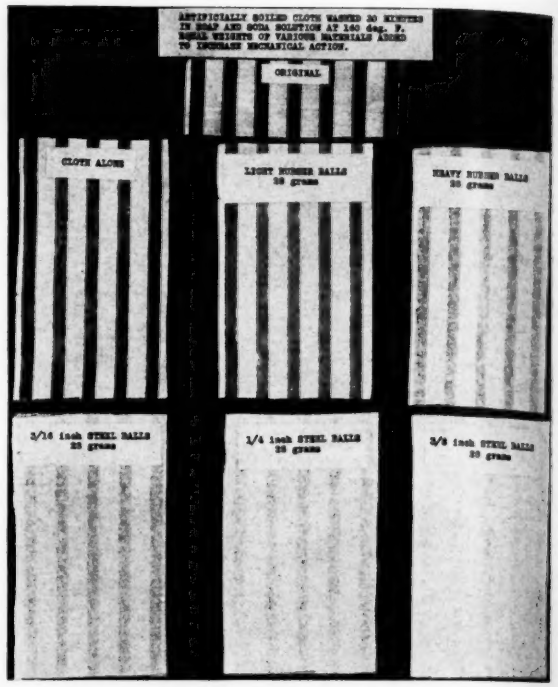


Fig. 6—*Effect of balls on the removal of dirt. Note: Figs. 6 and 7 show greater contrast between the soiled (dark) stripes and the white stripes on the cloth than the samples do.*

Ten rubber spheres 1 cm. in diameter and weighing approximately 1.5 grams each shall be placed in the jars when specified.

Unless otherwise specified, the following routine procedure shall be followed: The machine shall be in readiness at the temperature of the test. The jars containing the washing solution shall be preheated to the desired temperature; for example, by standing in a pan of hot water. The samples shall then be added to the jars, which shall be quickly closed and placed in the machine and the machine started. A wash or rinse shall be timed from when the machine is started to when it is stopped. At the proper time the machine shall be stopped, the jars quickly removed and emptied one at a time through a screen so as to recover the samples. The samples shall then be returned to the jars and covered with a fresh detergent solution for further treatment or with water for rinsing. Finally they shall be centrifuged to remove excess moisture.

Unless otherwise specified, drying shall be carried out in a current of air at room temperature, as before a fan. Sample shall dry in 20 minutes.

Ironing shall be considered a part of the washing procedure only when called for in the test. The samples shall be iron damp, as they come from the centrifuge with the iron at a suitable temperature.

The sequence of operations, washing, rinsing, centrifuging, drying or ironing, shall be carried out without interruption.

The foregoing proposals for standardizing tests for the fastness to washing of colored textiles are tentative. They are given in this form for discussion. Criticisms and suggestions for improving them will be appreciated.

ACKNOWLEDGMENT

The authors wish to acknowledge the valuable co-operation of George H. Johnson and the Laundryowners' National Association. They are indebted to the Atlas Electric Devices Company, of Chicago, for the gift of the machine shown in Figs. 4 and 5 and for helpful co-operation.

LOWELL TEXTILE ALUMNI ELECT NEW YORK OFFICERS

At a recent meeting of the New York Alumni of the Lowell Textile Institute Herbert R. Mauersberger '18, was elected president, L. S. Zisman, '20, vice-president, and Frank R. McGowan, '15, secretary-treasurer. Messrs. James W. Cox, Jr., and Wm. O. Jelleme, of the Pacific Mills, and president of the L. T. I. Alumni Association, were elected trustees. Charles H. Eames and Royal P. White, agent of the Sterling Mills, Lowell, Mass., were made honorary members.

Members of practically every class since 1900 were in attendance and enjoyed the banquet and entertainment, followed by a business meeting. The guests of the evening were Charles H. Eames, president of Lowell Textile Institute, and Prof. Arthur A. Stewart, Alumni Association. The former spoke inspiringly about the activities at Lowell, Mass. Professor Stewart reported on the progress of the Stephen E. Smith Scholarship Fund.

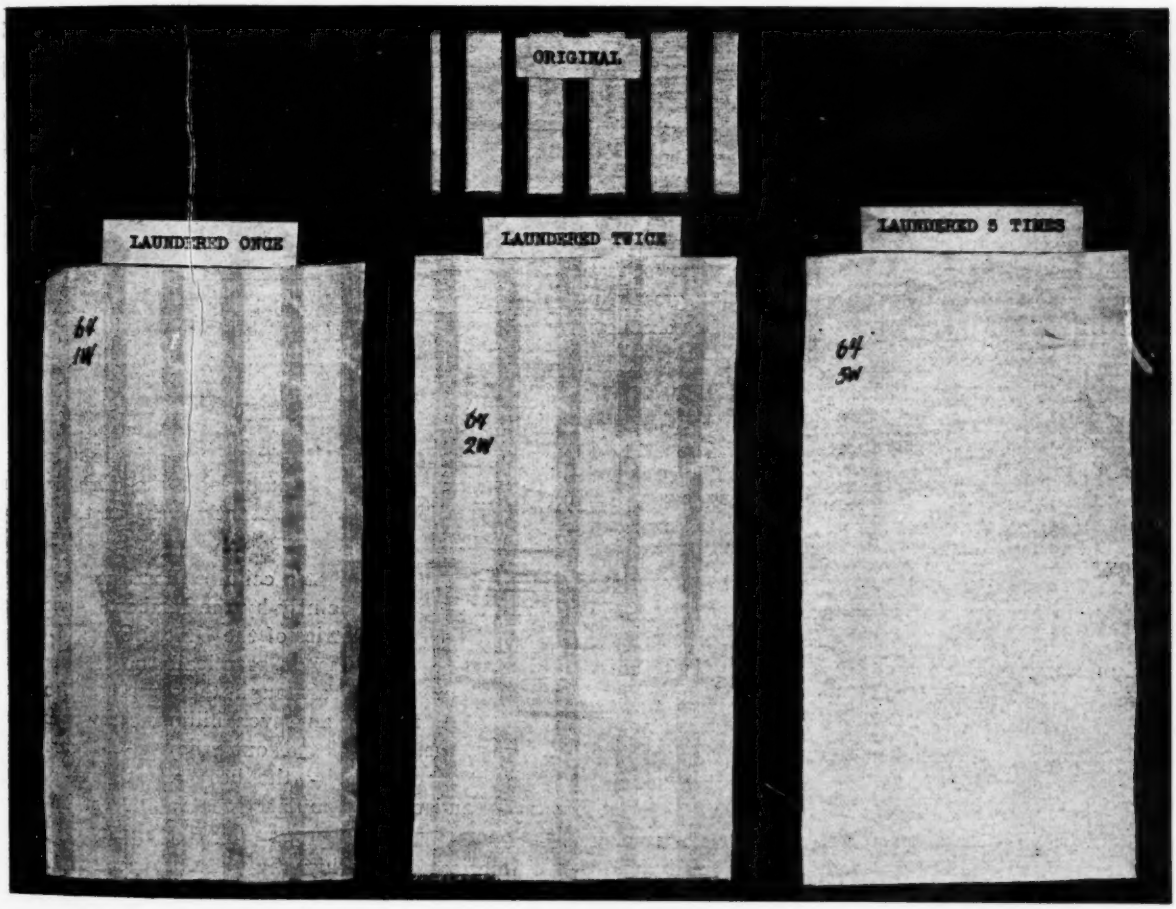


FIG. 7—*Effect of commercial laundering on cloth used for experiments shown in Fig. 6. A piece of the cloth, 12 by 30 inches in size, was washed with regular work in a commercial laundry by the formula for the "family bundle" (see Laundryowners' National Association "Manual of Standard Practice for the Power Laundry," Second Edition). The laundering procedure calls for one full hour in the commercial machine; the tests in Fig. 6 were washed only half an hour in the laboratory machine.*

Sericin and Fibroin

Advancing a Hypothesis That May Explain Differences in Silk

By E. W. Pierce

IN the American Dyestuff Repokter of September 3, 1928, there appeared an article by Fred. Grove-Palmer, A.I.C., discussing the solubility of sericin and advancing the current explanation of how a silk worm spins the silk fiber and coats it with gum—an explanation which everyone seems to take for granted without further inquiry. It is now ten years since I first advanced a hypothesis regarding this phenomenon, hoping research might be taken up by some who had the facilities of studying the living silkworm in a truly scientific way, such facilities being out of my reach.

The hypothesis will bear repetition. Aside from a simple dissection of the worm in different stages of growth and a chemical analysis of the internal secretion as well as the fiber and gum, little else has been done. Silk fiber may be named fibroin and the gum sericin, and they both may be referred to the great class of proteins and defined as amino acids with an accurate chemical formula, but we are still in the dark as to their physical form if we depend on chemistry only. Silk may be a chemical substance, but more than that it is a biological product, and its origin is closely bound up with obscure biological reactions which give rise to products of diverse properties without the change of the atomic relations shown in the chemical formula. Every chemist knows that many natural organic products, while differing greatly in chemical and physical properties, may be chemical isomerides.

The Silkworm's Sacs

The dissection of the silkworm reveals two sacs, one on each side, discharging into the spinning apparatus. The contents of these sacs are liquid and the material is neither fiber or gum, just a colloidal protein dispersion. There is no separate supply of sericin to coat the spun fiber. The contents of the sacs will not form silk fiber either by evaporation or by oxidation, and here the problem stands. There are, however, two tiny glands, whose function has never been determined, that are also connected with the spinning tubes. These glands are so small that their entire contents could not yield enough gum to coat one inch of silk fiber. Now my hypothesis is that the contents of the main sacs is a liquid which, as it exudes into the air, receives a very small amount of a coagulant or clothing material from the small glands. Just as blood, mixed with a trace of the liquids from the body cells, clots to form fibroin and serum, so the silk worm secretion clots to form fibroin and sericin. In the cocoon the ability to either evaporate or oxidize would steadily diminish, but by this bio-chemical reaction the fiber remains uniform from start to finish.

If this hypothesis be true it will also explain differences in the nature of silks. When the reaction is at its best the fiber is dense and hard, and there is plenty of gum, but when the coagulation is not perfect the fiber is not as dense and the gum is more difficult to remove. Such differences are probably due to a lack of certain mineral salts in the food of the worms from certain districts, first as a lack of calcium salts in the human body gives rise to the disease *hemophilia* or inability of the blood to clot.

The perfect silk fiber is no doubt structureless, but from its mode of formation it must retain internally a molecular arrangement that can become evident when it is gradually broken down by excessive action of alkalies. Just as the mineral or crystal, while homogeneous or flawless, will show cleavage planes when broken, and even fibrous structure, as in the case of asbestos, so the silk fiber will break down into fibrillae which roll up on themselves and form the well-known "silk louse." A simple liquid congealing into a fiber would not have this property, but would act exactly like the fibers of any of the well-known varieties of rayon, which are truly structureless internally.

It would be interesting to have this hypothesis confirmed by scientific research, but it is evident that it is a bit beyond the realm of the ordinary chemist or even the bio-chemist and furthermore requires a good supply of silkworms from different districts. Perhaps some of our Japanese friends will undertake the task.

DR. HERTY URGES STUDY OF CELLULOSE

Tells Engineers We Have Entered "Cellulose Age"

Scientific research on the properties and industrial uses of cellulose and its derivatives was strongly advocated by Dr. Charles H. Herty of the Chemical Foundation, Inc., in his address at the dinner of the Engineering Foundation recently held at the Union League Club, New York.

Dr. Herty urged that a cellulose institute be established and financed jointly by the lumber, cotton, textile, and paper industries of the United States. These industries are carrying on practically no technical research, he asserted, although they represent an investment of more than seven billions. In operating the institute, he pointed out, they could carry on productive research by the best talent that science affords, and could stimulate greater interest in cellulose chemistry among research groups in universities.

"America is entering the cellulose period," added Dr. Herty, addressing leading chemists and engineers from all over the country interested in research. "Forests, farms and gardens constitute a vast storehouse of cellulose, which when intelligently utilized through science and industry will add enormously to the nation's wealth."

Mercerization of Cotton With Sulphuric Acid

Part II

The Influencing Factors in Acid Mercerization—Discussion of Results—Relation of Various Factors to Time of Immersion, Tensile Strength, Extensibility, Etc.—Theoretical Discussion and Conclusions

(*Continued from page 649, October 15 issue.*)

By W. B. SELLARS and F. C. VILBRANDT

THE previous experiments established definite conditions and methods for controlling the factor studied in the sulphuric acid mercerization investigation. Runs were then made using acid concentrations of 30, 40, 50, 60, 62.5, 65 and 70% sulphuric acid. For each concentration runs were made at temperatures of 0 to 3°, 20°, 50° and either 65° or 80° C., according as the cotton would stand treatment at the higher temperature without disintegration. At each temperature runs were made both with and without application of tension, using the times of immersion selected in section (H). The results are given in tables III to IX and the accompanying curves (curves 1 to 12).

Discussion of Results

Relation of Tensile Strength to Time of Immersion (*Curves 1 to 4*).—With concentrations of 30 to 60% sulphuric acid and temperatures of 0 to 20° C. the tensile strength decreases slightly during the first ¼ minute of immersion, then remains practically constant through 5 minutes immersion. Above 20° C. the strength decreases on immersion 30 to 60% acid. Samples treated with tension show slightly greater strength at all temperatures than those treated without tension.

With concentrations of 60 to 66% acid at 0 to 20° C. there is an increase of strength of 20 to 25% when immersed for ½ minute. After ½ minute immersion the strength begins to decrease rapidly with increasing time of immersion. At temperatures above 20° the cotton is attacked immediately and loses strength.

With 70% acid there is a slight increase in strength with ¼ minute immersion under tension. Longer periods of time than this destroy the cotton very rapidly.

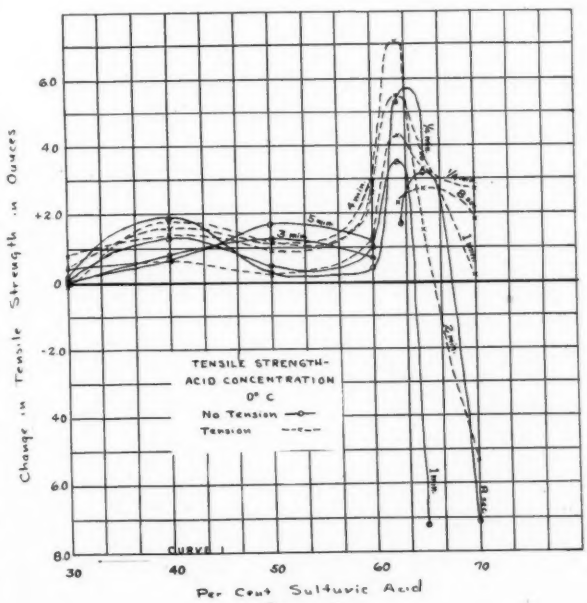
At all concentrations and temperatures the yarn can be immersed longer without a decrease in strength when under tension.

At concentrations above 60% the cotton cannot be immersed longer than 2 to 3 minutes without being gelatinized and finally dissolved.

Relation of Extensibility to Time of Immersion (*Curves 5 to 8*).—With concentrations of 30 to 60% sulphuric acid at temperatures of 0 to 50° C. the cotton gains 25 to 35% of its original extensibility during the first ¼ minute immersion, the gain then remaining fairly constant through 5 minutes immersion. Samples treated without tension generally gain about twice as much as those treated with tension. At temperatures higher than 50° the extensibility decreases immediately upon immersion.

With 60 to 70% acid at 0° C. and no tension, the extensibility increases enormously—e. g., from 4 to 12–30% for times of immersion up to 1 minute. With tension there is a rise of about 1% to a maximum at ¼ minute immersion, then a decrease. At higher temperatures the cotton will not stand treatment unless in the stretched condition.

Relation of Dye Affinity to Time of Immersion (*Curves 9 to 12*).—Time of immersion has little effect on the affinity for dye with concentrations below 50% sulphuric acid. At concentrations above this and at temperatures of 0 to 30° the dye affinity increases very rapidly during the first ¼ minute immersion, then changes very little for times of immersion through 5 minutes. The stronger the acid the greater the affinity for dye with these concentrations. At temperatures above 30° the affinity decreases continuously with increasing time. Samples



Curve 1

TABLE III—30% SULPHURIC ACID

No.	Temp. (° C.)	Stretch	Time of Immersion (Min.)	Affinity for Dye	Regain (P.C.)	Strength of Treated (Oz.)	Strength of Untreated (Oz.)	Change in Strength (Oz.)	Extens. of Treated (P.C.)	Extens. of Untreated (P.C.)	Change in Extens. (P.C.)	Shrinkage ±0.6% (P.C.)	Change in Shrinkage (P.C.)
A 1a	0	—	¼	0	4.7	17.9	17.7	+ 0.2	6.0	5.5	+ 0.5	1.9	0
1b	0	—	1	0	4.7	17.8	17.7	+ 0.1	6.1	5.5	+ 0.6	1.9	0
1c	0	—	3	0	4.7	17.7	17.7	0	6.2	5.5	+ 0.7	2.0	0
1d	0	—	5	0	4.7	17.8	17.7	+ 0.1	6.1	5.5	+ 0.6	1.2	— 0.2
2a	0	+	¼	0	4.7	18.0	17.6	+ 0.4	6.0	5.9	+ 0.1	2.1	0
2b	0	+	1	0	4.7	17.5	17.6	— 0.1	5.8	5.9	— 0.1	0.1	— 1.4
2c	0	+	2	0	5.2	18.6	17.8	+ 0.8	6.1	6.1	0	0.7	— 0.6
2d	0	+	4	0	5.2	18.2	17.8	+ 0.4	6.1	6.1	— 0.1	1.8	0
B 1a	20	—	¼	0	4.7	17.4	17.7	— 0.3	6.2	5.5	+ 0.7	3.7	+ 1.1
1b	20	—	1	0	4.7	18.6	17.7	+ 0.9	6.3	5.5	+ 0.8	0.8	— 0.6
1c	20	—	3	0	6.5	18.5	18.6	— 0.1	6.6	6.1	+ 0.5	2.1	0
1d	20	—	5	0	6.5	19.2	18.6	+ 0.6	6.7	6.1	+ 0.6	1.7	0
2a	20	+	¼	0	4.8	17.7	17.6	— 0.1	5.7	5.9	— 0.2	1.3	— 0.2
2b	20	+	1	0	4.8	17.7	17.6	— 0.1	5.5	5.9	— 0.4	0.5	— 1.0
2c	20	+	2	0	6.5	19.2	18.5	+ 0.7	6.5	6.5	0	0.4	— 1.1
2d	20	+	4	0	4.8	17.9	17.6	+ 0.3	6.0	5.9	+ 0.1	1.9	0
C 1a	20	—	¼	0	6.1	19.0	18.6	+ 0.4	6.9	6.0	+ 0.9	1.9	0
1b	20	—	1	0	6.1	19.3	18.6	+ 0.7	7.0	6.0	+ 1.0	1.9	0
1c	20	—	3	0	6.1	18.2	18.6	— 0.4	6.9	6.0	+ 0.9	2.1	0
1d	20	—	5	0	6.1	17.3	18.6	— 1.3	6.8	6.0	+ 0.8	1.8	0
2a	20	+	¼	0	6.1	20.0	18.5	+ 1.5	6.9	6.4	+ 0.5	1.4	0
2b	20	+	1	0	6.1	19.0	18.5	+ 0.5	6.8	6.4	+ 0.4	1.6	0
2c	20	+	2	0	6.1	19.0	18.5	+ 0.5	6.7	6.4	+ 0.3	2.1	0
2d	20	+	4	0	6.1	16.4	18.5	— 2.1	5.9	6.4	— 0.5	1.9	0
D 1a	80	—	¼	a	6.6	16.1	18.7	— 2.6	6.3	6.1	+ 0.2	1.8	0
1b	80	—	1	a—	6.6	13.0	18.7	— 5.7	5.4	6.1	— 0.7	1.9	0
1c	80	—	3	b+	6.6	7.6	18.7	—11.1	3.7	6.1	— 2.4	3.4	+ 0.8
1d	80	—	5	b	6.6	5.2	18.7	—13.5	2.8	6.1	— 3.3	1.2	— 0.2
2a	80	+	¼	a+	6.6	16.0	18.6	— 2.6	5.8	6.5	— 0.7	0	— 1.5
2b	80	+	1	a	6.6	13.1	18.6	— 5.5	4.8	6.5	— 1.7	0	— 1.5
2c	80	+	2	a	6.6	10.6	18.6	— 8.0	4.2	6.5	— 2.3	1.4	— 0.1
2d	80	+	4	(*)	6.6	3.2	18.6	—15.4	2.0	6.5	— 4.5	...	...

*Disintegrated so badly that skeins could not be used for measuring shrinkage and determination of dye affinity.

LUSTER

A-1. Slightly improved; whiter; threads larger.

A-2. Same as A-1, but threads not as large.

B-1 and B-2. Very slightly increased luster; all threads apparently larger; stretched smaller than unstretched.

C-1. Very slight increase.

C-2. A slightly greater increase than in C-1; no variation between a, b, c and d.

D-1. Very slightly increased.

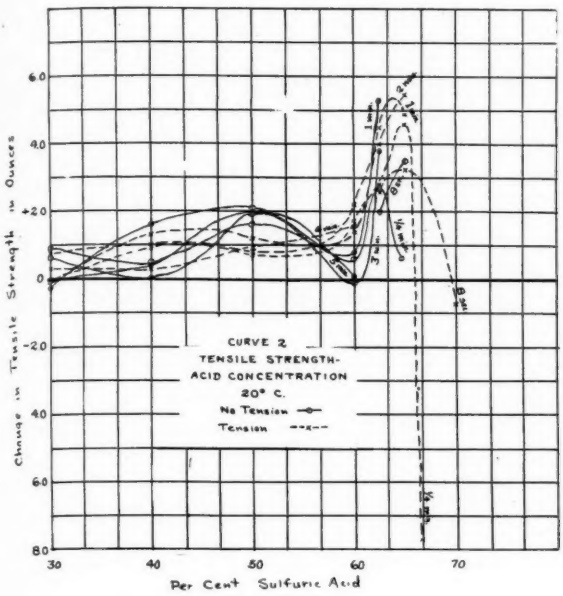
D-2. Better than D-1.

treated without tension show a greater affinity for dye than those treated with tension.

Effect of Temperature on Tensile Strength (*Curves 1 to 4*).—The temperature at which the yarn can be immersed without showing a loss of strength decreases with rising acid concentration. For 30% acid this temperature is 50°C.; for 40 to 50% acid, 35°, and for 60% acid, 20°.

For concentrations up to 60% there is little or no increase in strength at low temperatures. However, with temperatures of 0 to 30° with 62.5 to 65.0% acid a greatly increased strength results. The curves for 62.5% acid indicate that the strength reaches a maximum value at some temperature below 0° C.; at this temperature the strength is decreasing. With 65% acid a maximum strength is obtained with short periods of immersion and with tension at 20°. However, this increase is not as great as that obtained at 0° with 62.5% acid.

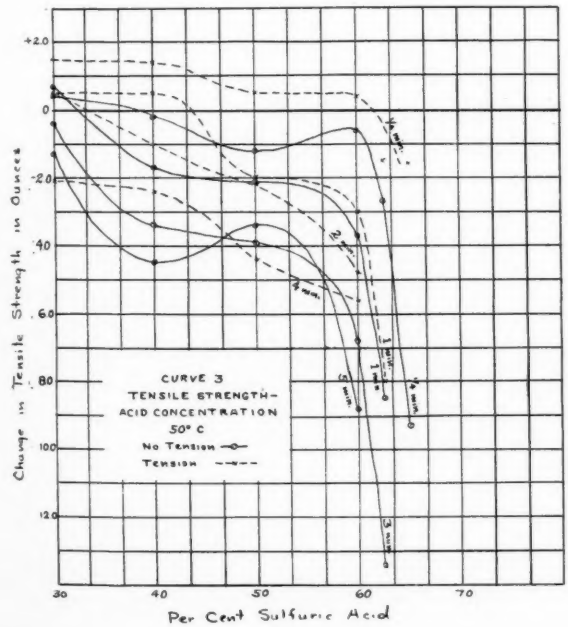
Effect of Temperature of Acid on Extensibility (*Curves 5 to 8*).—With concentrations below 60% sulphuric acid and without tension the extensibility is increased practically the same amount for all temperatures below 50° C. Acid below 50% in concentration has no effect upon the extensibility of samples treated with tension at tempera-



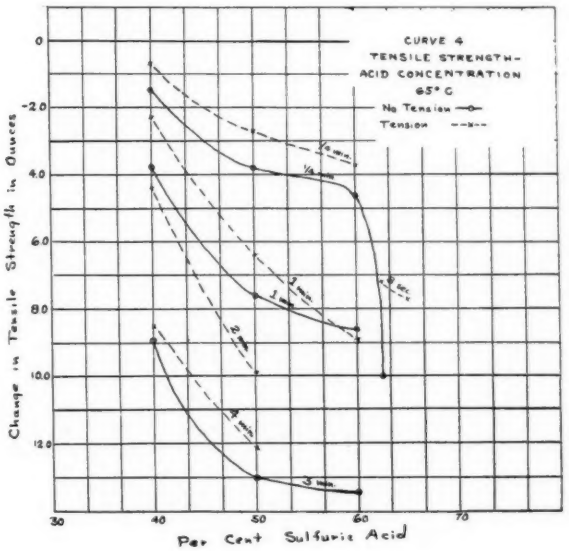
tures below 50° C. At higher temperatures the extensibility decreases rapidly with increasing temperature, both with and without tension.

With 60 to 65% acid at temperatures below 20° and without tension there is a very great increase in the extensibility, with indication of a maximum increase at or slightly below 0° with 62.5% acid.

Effect of Temperature of Acid on Dye Affinity (*Curves* 9 *to* 12).—At low concentrations—viz., 40 to 50%—increasing temperature decreases the affinity for dye; 30% acid has no effect upon the dye affinity except at temperatures near 80°, which decrease the dye affinity. With 60 to 65% acid there is evidence of a maximum in the curve below 0°, where the dye affinity is greatly increased. From this temperature upward it decreases gradually with increasing temperature until at 40° it shows practically the same dye affinity as the untreated cotton. With 65% acid, no tension and very short periods of immersion, this maximum affinity for dye is obtained at 25°.



Effect of Acid Concentration on Tensile Strength (*Curves* 1 *to* 4).—At temperatures of 0 to 20° C. concentrations below 60% have very little effect on the strength with times of immersion of 8 seconds to 5 minutes. With 60% acid, however, the strength begins to increase very much, reaching a maximum value with 62.5% acid, then decreasing rapidly again with rising concentration, reaching the value of its normal strength at a concentration of about 65%. At concentrations



above 65% it first swells greatly, gelatinizing, then disintegrates and dissolves.

At temperatures of 50 to 65° these maximum values of strength obtained with lower temperatures have disappeared and instead of a gain in strength there is a loss, which becomes very great with concentrations of 60% and above.

Effect of Concentration of Acid on Extensibility (*Curves* 5 *to* 8).—In the case of yarn treated without tension the same can be said of the effect of acid concentration on extensibility as was said for its effect upon the tensile strength: There is a sharp maximum in the curve at a concentration of 62.5% acid at temperatures of 0 to 20°, the greatest increase in extensibility being obtained at this point. However, in this case the yarn begins to disintegrate and dissolve before the extensibility has fallen back to its normal value as the acid concentration increases. With yarn treated under tension there is only a slight increase in extensibility at temperatures of 0 to 20°; there is a poorly defined maximum gain

TABLE IV—40% SULPHURIC ACID

No.	Temp. (° C.)	Stretch	Time of Immersion (Min.)	Affinity for Dye	Regain (P.C.)	Strength of Treated (Oz.)	Strength of Untreated (Oz.)	Change in Strength (Oz.)	Extens. of Treated (P.C.)	Extens. of Untreated (P.C.)	Change in Extens. (P.C.)	Shrinkage ±0.6% (P.C.)	Change in Shrinkage (P.C.)
A 1a	0	—	¼	0	5.9	20.2	18.3	+ 1.9	7.5	5.9	+ 1.6	0.7	— 0.7
1b	0	—	1	0	5.9	19.6	18.3	+ 1.3	7.3	5.9	+ 1.4	1.5	0
1c	0	—	3	0	5.9	19.1	18.3	+ 0.8	7.2	5.9	+ 1.3	1.7	0
1d	0	—	5	0	5.9	19.0	18.3	+ 0.7	7.0	5.9	+ 1.1	1.5	0
2a	0	+	¼	0	5.5	18.7	18.0	+ 0.7	6.3	6.2	+ 0.1	...	...
2b	0	+	1	0	5.5	19.8	18.0	+ 1.8	6.7	6.2	+ 0.5	...	...
2c	0	+	2	0	5.5	19.4	18.0	+ 1.4	6.7	6.2	+ 0.5	...	...
2d	0	+	4	0	5.5	19.6	18.0	+ 1.6	6.6	6.2	+ 0.4	...	...
B 1a	20	—	¼	0	6.5	20.2	18.6	+ 1.6	7.6	6.1	+ 1.5	1.5	0
1b	20	—	1	0	6.5	19.1	18.6	+ 0.5	7.3	6.1	+ 1.2	0.9	— 0.5
1c	20	—	3	0	6.5	19.0	18.6	+ 0.4	7.4	6.1	+ 1.3	2.0	0
1d	20	—	5	0	6.5	18.6	18.6	0	7.3	6.1	+ 1.2	2.0	0
2a	20	+	¼	0	6.1	19.2	18.3	+ 0.9	6.7	6.4	+ 0.3	...	...
2b	20	+	1	0	6.1	19.6	18.3	+ 1.3	6.9	6.4	+ 0.5	...	...
2c	20	+	2	0	6.1	19.8	18.3	+ 1.5	6.5	6.4	+ 0.1	...	...
2d	20	+	4	0	6.1	18.6	18.3	+ 0.3	6.6	6.4	+ 0.2	...	...
C 1a	50	—	¼	0	5.0	17.7	17.9	— 0.2	7.0	5.6	+ 1.4	1.7	0
1b	50	—	1	0—	5.0	16.2	17.9	— 1.7	6.7	5.6	+ 1.1	1.6	0
1c	50	—	3	0—	5.0	14.5	17.9	— 3.4	6.2	5.6	+ 0.6	1.3	— 0.1
1d	50	—	5	a+	5.0	13.4	17.9	— 4.5	5.9	5.6	+ 0.3	1.8	0
2a	50	+	¼	0—	5.0	19.2	17.8	+ 1.4	5.3	6.0	— 0.7	...	...
2b	50	+	1	0—	5.0	18.3	17.8	+ 0.5	5.5	6.0	— 0.5	...	...
2c	50	+	2	a+	5.0	16.8	17.8	— 1.0	5.3	6.0	— 0.7	...	...
2d	50	+	4	a	5.0	15.4	17.8	— 2.4	4.5	6.0	— 1.5	...	...
D 1a	65	—	¼	0—	5.0	16.4	17.9	— 1.5	5.4	5.6	— 0.2	2.4	0
1b	65	—	1	a+	5.0	14.1	17.9	— 3.8	5.0	5.6	— 0.6	2.0	0
1c	65	—	3	a—	5.0	9.0	17.9	— 8.9	3.8	5.6	— 1.8	2.8	+ 0.2
1d	65	—	5	b+	5.0	8.1	17.9	— 9.8	3.6	5.6	— 2.0	2.8	+ 0.2
2a	65	—	¼	a+	5.0	17.1	17.8	— 0.7	5.4	6.0	— 0.6	...	...
2b	65	—	1	a	5.0	15.5	17.8	— 2.3	4.6	6.0	— 1.3	...	...
2c	65	—	2	a—	5.0	13.4	17.8	— 4.4	4.0	6.0	— 2.0	...	...
2d	65	—	4	b	5.0	9.3	17.8	— 8.5	3.3	6.0	— 2.7	...	...

LUSTER

A-1 and A-2. No change. B-1 and B-2. No change. C-1 and C-2. No change. D-1 and D-2. No change.

in extensibility at a concentration of approximately 53% sulphuric acid.

At 50° C. there is only a slight trace of this maximum point obtained with 62.5% acid at lower temperatures on yarn treated without tension, and at 65° it has disappeared entirely.

At 50 to 65° C. yarn treated under tension shows a decrease with all concentrations of acid used.

Effect of Acid Concentration on Affinity for Dyes (Curves 9 to 12).—At 0 to 20° C. concentrations below 55% sulphuric acid have little effect on the affinity for dye. With concentrations of 60 to 70% the dye affinity increases very much, showing a maximum increase in affinity with acid of approximately 65% sulphuric acid.

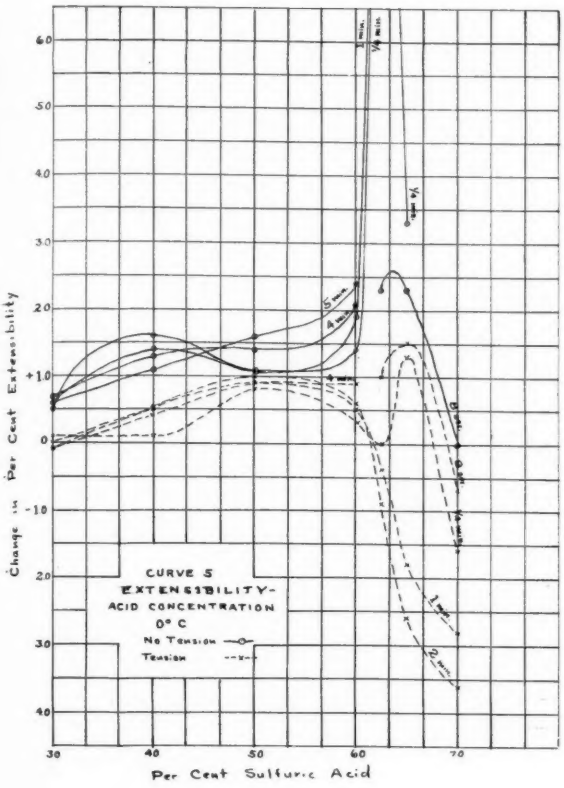
At higher temperatures all concentrations of acid above 30% decrease the affinity for dye; with 60% acid this decrease becomes very great.

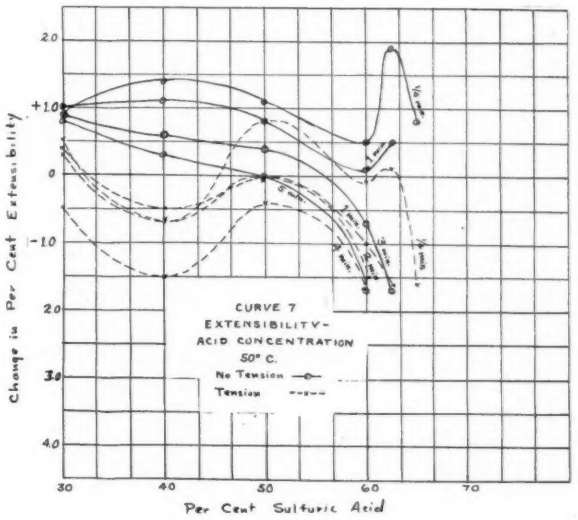
Effect of Acid on Shrinkage

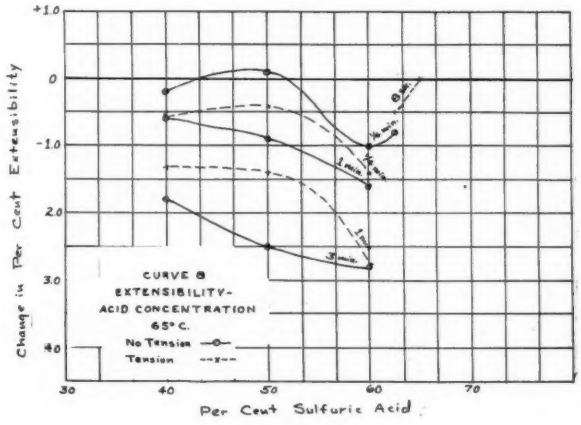
With 60% sulphuric acid and all concentrations below this, the shrinkage results were very irregular where any change at all occurred, and indicate nothing.

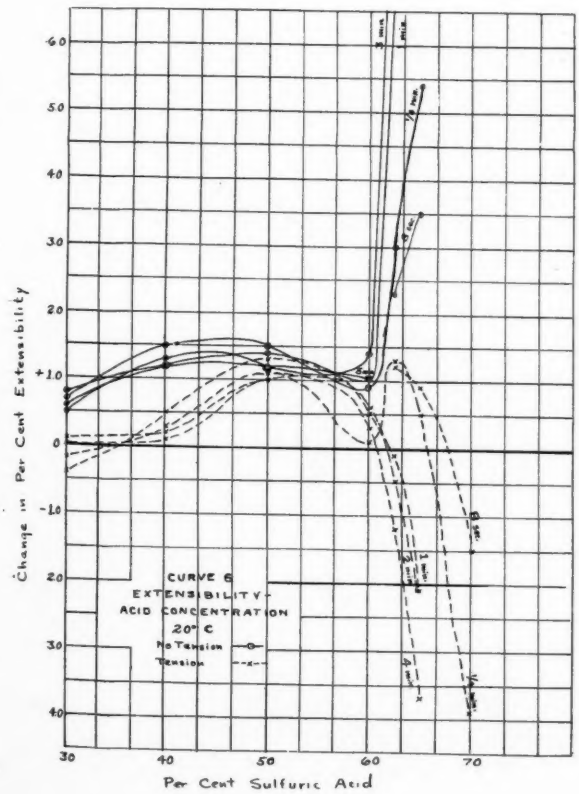
With concentrations of 62.5 to 70% sulphuric acid, samples treated without tension at temperatures of 0 to 50° C. show a marked shrinkage which becomes very great with 70% acid, the yarn shrinking to such an extent that it cannot be unraveled to remeasure if immersed longer than ¼ minute. Increase of the time of immersion increases the amount of shrinkage.

On the other hand, samples of yarn treated at these concentrations and temperatures on the stretching forms become loose and increase in length, the maximum increase observed being 4.2%.


CURVE 5
EXTENSIBILITY-
ACID CONCENTRATION
0° C
No Tension
Tension
Change in Per Cent Extensibility
Per Cent Sulfuric Acid


CURVE 7
EXTENSIBILITY-
ACID CONCENTRATION
50° C.
No Tension
Tension
Change in Per Cent Extensibility
Per Cent Sulfuric Acid


CURVE 8
EXTENSIBILITY-
ACID CONCENTRATION
65° C.
No Tension
Tension
Change in Per Cent Extensibility
Per Cent Sulfuric Acid


CURVE 6
EXTENSIBILITY-
ACID CONCENTRATION
20° C
No Tension
Tension
Change in Per Cent Extensibility
Per Cent Sulfuric Acid

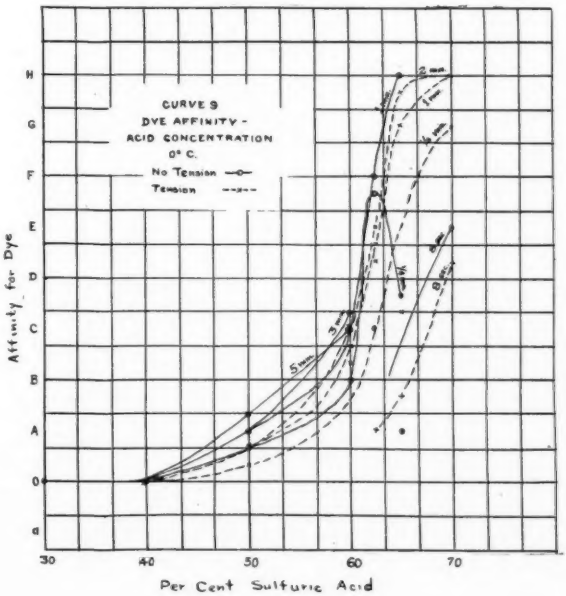

CURVE 9
DYE AFFINITY -
ACID CONCENTRATION
0° C.
No Tension
Tension
Affinity for Dye
Per Cent Sulfuric Acid

TABLE V—50% SULPHURIC ACID

No.	Temp. (° C.)	Stretch	Time of Immersion (Min.)	Affinity for Dye	Regain (P.C.)	Strength of Treated (Oz.)	Strength of Untreated (Oz.)	Change in Strength (Oz.)	Extens. of Treated (P.C.)	Extens. of Untreated (P.C.)	Change in Extens. (P.C.)	Shrinkage ±0.6% (P.C.)	Change in Shrinkage (P.C.)
A 1a	0	—	¼	A—	4.5	17.9	17.6	+ 0.3	6.5	5.4	+ 1.1	...	...
1b	0	—	1	A	4.5	18.1	17.6	+ 0.5	6.5	5.4	+ 1.1	1.5	0
1c	0	—	3	A	5.9	19.7	18.4	+ 1.3	7.3	5.9	+ 1.4	2.1	0
1d	0	—	5	A+	5.9	20.1	18.4	+ 1.7	7.5	5.9	+ 1.6	1.5	0
2a	0	+	¼	O+	5.2	18.2	17.9	+ 0.3	6.9	6.1	+ 0.8	...	...
2b	0	+	1	A—	5.2	19.1	17.9	+ 1.2	7.0	6.1	+ 0.9	...	...
2c	0	+	2	A—	5.2	18.8	17.9	+ 0.9	7.1	6.1	+ 1.0	...	...
2d	0	+	4	A—	5.2	19.1	17.9	+ 1.2	7.0	6.1	+ 0.9	...	...
B 1a	20	—	¼	A—	5.2	20.1	18.0	+ 2.1	7.2	5.7	+ 1.5	1.5	0
1b	20	—	1	O—	5.2	19.6	18.0	+ 1.6	7.1	5.7	+ 1.4	1.9	0
1c	20	—	3	O+	5.2	20.0	18.0	+ 2.0	6.9	5.7	+ 1.2	2.0	0
1d	20	—	5	O+	5.2	20.0	18.0	+ 2.0	6.9	5.7	+ 1.2	2.3	0
2a	20	+	¼	O	4.5	18.2	17.5	+ 0.7	6.9	5.8	+ 1.1	...	...
2b	20	+	1	O	4.5	18.7	17.5	+ 1.2	7.1	5.8	+ 1.3	...	...
2c	20	+	2	O+	4.5	18.3	17.5	+ 0.8	6.8	5.8	+ 1.0	...	...
2d	20	+	4	O+	4.5	18.4	17.5	+ 0.9	6.8	5.8	+ 1.0	...	...
C 1a	50	—	¼	O—	4.5	16.4	17.6	— 1.2	6.5	5.4	+ 1.1	1.5	0
1b	50	—	1	a	4.5	15.5	17.6	— 2.1	6.2	5.4	+ 0.8	1.4	0
1c	50	—	3	a—	4.5	13.7	17.6	— 3.9	5.8	5.4	+ 0.4	2.1	0
1d	50	—	5	b+	4.5	14.2	17.6	— 3.4	5.4	5.4	0	2.0	0
2a	50	+	¼	O	4.5	18.0	17.5	+ 0.5	6.6	5.8	+ 0.8	...	...
2b	50	+	1	a	4.5	15.5	17.5	— 2.0	5.8	5.8	0	...	...
2c	50	+	2	a	4.5	15.4	17.5	— 2.1	5.8	5.8	0	...	...
2d	50	+	4	a—	4.5	13.1	17.5	— 4.4	5.4	5.8	— 0.4	...	...
D 1a	65	—	¼	a—	4.5	13.8	17.6	— 3.8	5.5	5.4	+ 0.1	1.2	— 0.2
1b	65	—	1	b—	4.5	10.0	17.6	— 7.6	4.5	5.4	— 0.9	—0.5*	— 1.9
1c	65	—	3	d	4.5	4.6	17.6	—13.0	2.9	5.4	— 2.5	1.2	— 0.2
1d	65	—	5	e+	4.5	4.6	17.6	—13.0	3.1	5.4	— 2.3	1.1	— 0.3
2a	65	+	¼	a—	5.7	15.4	18.1	— 2.7	5.8	6.2	— 0.4	...	...
2b	65	+	1	b	5.7	11.7	18.1	— 6.4	4.8	6.2	— 1.4	...	...
2c	65	+	2	c	5.7	8.2	18.1	— 9.9	3.8	6.2	— 2.4	...	...
2d	65	+	4	d	5.7	6.0	18.1	—12.1	3.4	6.2	— 2.8	...	...
E 1a	80	—	¼	c	4.5	8.1	17.6	— 9.5	3.8	5.4	— 1.6	1.3	— 0.1
1b	80	—	1	e	4.5	3.9	17.6	—13.7	2.7	5.4	— 2.7	0.4	— 1.0
1c	80	—	3	Disintegrated		...	...	...	...	...	...	...	...
1d	80	—	5	...	...	...	...	...	...	...	...	...	...
2a	80	+	¼	c+	4.8	9.8	17.6	— 7.8	4.2	5.9	— 1.7	...	...
2b	80	+	1	e	4.8	4.3	17.6	—13.3	2.6	5.9	— 3.3	...	...
2c	80	+	2	Disintegrated		...	...	...	...	...	...	...	...
2d	80	+	4	...	...	...	...	...	...	...	...	...	...

*Extended instead of shrunk.

NOTES ON LUSTER

A-1. Slightly inferior to untreated; probably due to the swelling.

A-2. Same as A-1.

B-1. No change; better than A-1.

B-2. Slightly increased luster, fairly good.

C-1 and C-2. Little change; no tension better than samples with tension.

D-1 and D-2. No change; stretched and unstretched about same. All turned very slightly brown.

E-1 and E-2. Inferior to untreated; E-1 slightly better than E-2. All browned very slightly.

Luster

With concentrations of 60% sulphuric acid and below, the acid has little effect upon the luster. High temperatures tend to give a cream coloration to the cotton. Those treated without tension swelled and appeared to have a much larger diameter than those treated with tension, which did not swell so much. For this reason they sometimes appeared to have a slightly better luster than those treated with tension and the untreated cotton; this was the case with 30% sulphuric acid.

With 62.5 and 65.0% acid an improved luster was

TABLE VI—60% SULPHURIC ACID

No.	Temp. (° C.)	Stretch	Time of Immersion (Min.)	Affinity for Dye	Regain (P.C.)	Strength of Treated (Oz.)	Strength of Untreated (Oz.)	Change in Strength (Oz.)	Extens. of Treated (P.C.)	Extens. of Untreated (P.C.)	Change in Extens. (P.C.)	Shrinkage ±0.6% (P.C.)	Change in Shrinkage (P.C.)
A 1a	0–5	—	¼	B	4.7	18.1	17.7	+ 0.4	6.9	5.5	+ 1.4	1.5	0
1b	0–5	—	1	C	4.7	18.7	17.7	+ 1.0	7.4	5.5	+ 1.9	1.5	0
1c	0–5	—	3	C+	4.7	18.4	17.7	+ 0.7	7.6	5.5	+ 2.1	1.5	0
1d	0–5	—	5	C	4.7	18.9	17.7	+ 1.2	7.9	5.5	+ 2.4	2.4	0
2a	0–5	+	¼	B+	4.5	18.8	17.5	+ 1.3	6.1	5.8	+ 0.3	2.4	0
2b	0–5	+	1	C—	4.5	20.1	17.5	+ 2.6	6.3	5.8	+ 0.5	1.9	0
2c	0–5	+	2	C	4.5	20.2	17.5	+ 2.7	6.4	5.8	+ 0.6	0.8	— 0.7
2d	0–5	+	4	C	4.5	20.4	17.5	+ 2.9	6.7	5.8	+ 0.9	0.9	— 0.6
B 1a	20	—	¼	A	5.4	18.7	18.1	+ 0.6	6.6	5.7	+ 0.9	2.6	0
1b	20	—	1	C	5.5	18.9	18.1	+ 0.8	6.8	5.8	+ 1.0	3.6	+ 1.0
1c	20	—	3	C	5.5	18.0	18.1	— 0.1	7.2	5.8	+ 1.4	4.5	+ 1.9
1d	20	—	5	B—	5.5	18.2	18.1	+ 0.1	6.8	5.8	+ 1.0	2.1	0
2a	20	+	¼	A	4.7	19.3	17.6	+ 1.7	6.1	5.9	+ 0.2	2.5	0
2b	20	+	1	A+	4.7	19.0	17.6	+ 1.4	6.4	5.9	+ 0.5	2.7	0
2c	20	+	2	B—	4.7	19.8	17.6	+ 2.2	6.5	5.9	+ 0.6	1.5	0
2d	20	+	4	A+	4.7	19.2	17.6	+ 1.6	6.2	5.9	+ 0.3	1.6	0
C 1a	50	—	¼	O	5.2	17.4	18.0	— 0.6	6.2	5.7	+ 0.5	2.4	0
1b	50	—	1	a	5.2	14.3	18.0	— 3.7	5.8	5.7	+ 0.1	2.7	+ 0.1
1c	50	—	3	b	5.2	11.2	18.0	— 6.8	5.0	5.7	— 0.7	2.8	+ 0.2
1d	50	—	5	c	5.2	9.2	18.0	— 8.8	4.0	5.7	— 1.7	2.4	0
2a	50	+	¼	O—	5.2	18.3	17.9	+ 0.4	6.0	6.1	— 0.1	...	...
2b	50	+	1	a—	5.2	14.9	17.9	— 3.0	5.1	6.1	— 1.0	...	...
2c	50	+	2	b—	5.2	13.1	17.9	— 4.8	4.6	6.1	— 1.5	...	...
2d	50	+	4	c+	5.2	12.3	17.9	— 5.6	4.4	6.1	— 1.7	...	...
D 1a	65	—	¼	a—	5.5	13.5	18.1	— 4.6	4.8	5.8	— 1.0	2.4	0
1b	65	—	1	b—	5.2	9.4	18.0	— 8.6	4.1	5.7	— 1.6	1.7	0
1c	65	—	3	d	5.2	4.6	18.0	—13.4	2.9	5.7	— 2.8	1.8	0
1d	65	—	5	Disintegrated		...	...	...	...	...	...	...	...
2a	65	+	¼	a—	5.5	14.3	18.0	— 3.7	4.8	6.2	— 1.4	...	...
2b	65	+	1	d+	5.5	9.1	18.0	— 8.9	3.4	6.2	— 2.8	...	...
2c	65	+	2	Disintegrated		...	...	...	...	...	...	...	...
2d	65	+	4	Disintegrated		...	...	...	...	...	...	...	...
E 1a	80	—	¼	d	5.5	6.9	18.1	—11.2	3.3	5.8	— 2.5	2.7	+ 0.1
1b	80	—	1	Disintegrated		...	...	...	...	...	...	...	...
1c	80	—	3	Disintegrated		...	...	...	...	...	...	...	...
1d	80	—	5	Disintegrated		...	...	...	...	...	...	...	...
2a	80	+	¼	Disintegrated		...	...	...	...	...	...	...	...
2b	80	+	1	Disintegrated		...	...	...	...	...	...	...	...
2c	80	+	2	Disintegrated		...	...	...	...	...	...	...	...
2d	80	+	4	Disintegrated		...	...	...	...	...	...	...	...

Luster

A-1 and A-2. Whiter; little change in luster except that due to swelling of the threads. (False luster.)

B-1 and B-2. Not as good as A-1 and A-2.

C-1 and C-2. Practically no change; C-2 better than C-1.

D-1 and D-2. Apparently better, due to swelling of threads. D-1 better than D-2; better with increasing time of immersion.

E-1. Slightly better than D-1a.

TABLE VII—62.5% SULPHURIC ACID

No.	Temp. (° C.)	Stretch	Time of Immersion	Affinity for Dye	Regain (P.C.)	Strength of Treated (Oz.)	Strength of Untreated (Oz.)	Change in Strength (Oz.)	Extens. of Treated (P.C.)	Extens. of Untreated (P.C.)	Change in Extens. (P.C.)	Shrinkage ±0.6% (P.C.)	Change in Shrinkage (P.C.)
A 1a	0–2.5	—	8 sec.	C	5.5	19.8	18.1	+ 1.7	8.1	5.8	+ 2.3	0.7	— 0.7
1b	0–2.5	—	¼ min.	F—	5.5	23.4	18.1	+ 5.3	16.6	5.8	+10.8	14.9	+12.3
1c	0–2.5	—	1 min.	F	5.5	21.6	18.1	+ 3.5	18.7	5.8	+12.9	19.8	+17.2
1d	0–2.5	—	3 min.	Shrunk to harsh, stiff mass.				...	...	...	...	...	...
2a	0–2.5	+	8 sec.	A	5.5	20.3	18.0	+ 2.3	7.2	6.2	+ 1.0	1.8	0
2b	0–2.5	+	¼ min.	D+	5.5	22.3	18.0	+ 4.3	6.2	6.2	0	0.7	— 0.8
2c	0–2.5	+	1 min.	E—	5.5	23.5	18.0	+ 5.5	5.8	6.2	— 0.4	1.2	— 0.3
2d	0–2.5	+	2 min.	E	5.5	25.1	18.0	+ 7.1	5.3	6.2	— 0.9	0	— 1.5
B 1a	20	—	8 sec.	A	9.0	21.9	19.9	+ 2.0	9.3	7.0	+ 2.3	1.7	0
1b	20	—	¼ min.	B	9.0	22.6	19.9	+ 2.7	10.0	7.0	+ 3.0	2.7	0
1c	20	—	1 min.	D	9.0	25.2	19.9	+ 5.3	14.2	7.0	+ 7.2	7.1	+ 4.5
1d	20	—	3 min.	D—	9.0	23.7	19.9	+ 3.8	14.3	7.0	+ 7.3	7.8	+ 5.2
2a	20	+	8 sec.	A+	9.0	22.4	19.8	+ 2.6	8.6	7.4	+ 1.2	1.7	0
2b	20	+	¼ min.	C	9.0	22.6	19.8	+ 2.8	8.7	7.4	+ 1.3	1.1	— 0.4
2c	20	+	1 min.	D+	9.0	24.3	19.8	+ 4.5	7.3	7.4	— 0.1	Ext.0.6	— 2.1
2d	20	+	2 min.	D	9.0	23.8	19.8	+ 4.0	6.9	7.4	— 0.5	Ext.1.2	— 2.7
2e	20	+	4 min.	D—	5.6	22.0	18.0	+ 4.0	5.0	6.2	— 1.2	0.2	— 1.3
C 1a	50	—	8 sec.	O+	5.6	15.8	18.1	— 2.3	7.4	5.8	+ 1.6	2.8	+ 0.2
1b	50	—	¼ min.	a	5.6	15.4	18.1	— 2.7	7.7	5.8	+ 1.9	3.7	+ 1.1
1c	50	—	1 min.	d+	5.6	9.6	18.1	— 8.5	6.3	5.8	+ 0.5	4.7	+ 2.1
1d	50	—	3 min.	e+	5.6	4.7	18.1	—13.4	4.1	5.8	— 1.7	4.0	+ 1.4
2a	50	+	8 sec.	A—	5.6	17.7	18.0	— 0.3	6.9	6.2	+ 0.7	0.8	— 0.7
2b	50	+	¼ min.	a+	5.6	16.5	18.0	— 1.5	6.3	6.2	+ 0.1	1.6	0
2c	50	+	1 min.	d—	5.6	10.0	18.0	— 8.0	4.6	6.2	— 1.6	2.1	0
2d	50	+	2 min.	Threads broke			...	...	...	...	...	...	...
D 1a	65	—	8 sec.	c—	5.6	9.9	18.1	— 8.2	5.3	5.8	— 0.5	2.5	0
1b	65	—	¼ min.	d	5.6	8.1	18.1	—10.0	5.0	5.8	— 0.8	3.1	+ 0.5
1c	65	—	1 min.	Disintegrated (th'ds broke)				...	...	...	...	...	...
1d	65	—	3 min.	Disintegrated (th'ds broke)				...	...	...	...	...	...
2a	65	+	8 sec.	c	5.6	10.8	18.0	— 7.2	4.8	6.2	— 1.4	1.1	— 0.4
2b	65	+	¼ min.	Threads broke			...	...	...	...	...	...	...
2c	65	+	1 min.	Threads broke			...	...	...	...	...	...	...
2d	65	+	2 min.	Threads brone			...	...	...	...	...	...	...

LUSTER

A-1. a, very slight improvement; b and c, progressive decrease to no luster at all.

A-2. a, no change; b, c and d improved progressively; not quite as good as B-2.

B-1. a and b, very slight increase, others decreasing progressively.

B-2. Best improvement in luster obtained; a, no change; b and c best, and d, slight improvement; d slightly stiff in feel; e, decreased luster; luster retained after dyeing.

C-1. No change; slight decrease in c and d.

C-2. All slightly improved; a whiter than c.

D-1. No change.

D-2. Very slight improvement.

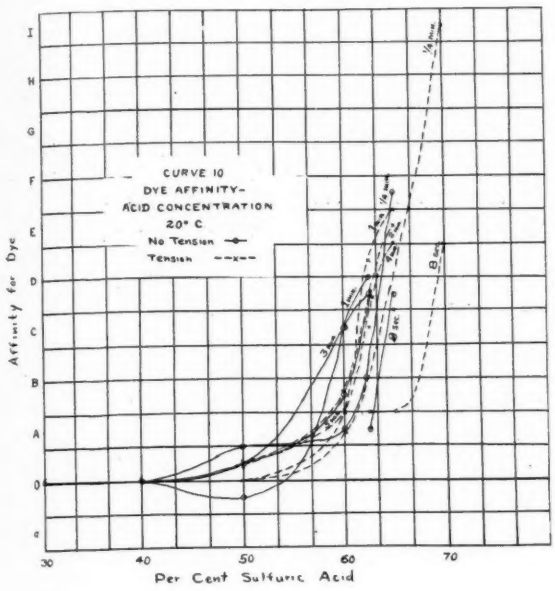
---

obtained with samples treated at 0 to 20° C., under tension. The best results were obtained with 62.5% acid at 20° C., immersing under tension from ¼ to 2 minutes. With greater times of immersion the cotton swells very much and becomes transparent while in the acid, but on washing out the acid with water it becomes opaque white. When dried, this product is stiff and straw-like. It also has a very great affinity for dye.

The mercerized product obtained with 62.5% sulphuric acid has only approximately two-thirds the luster which cotton mercerized by sodium hydroxide has. It retains its increased luster on dyeing. It also has a rather harsh "feel" which is still retained after dyeing; this harshness is a direct function of the time of immersion in the acid.

Samples treated with 62.5 to 65.0% acid at 0 to 20° C. without tension show a slight increase in luster over untreated cotton, but do not compare with those treated under tension in luster.

Under the microscope, those samples treated with tension as above and having an increased luster are seen to



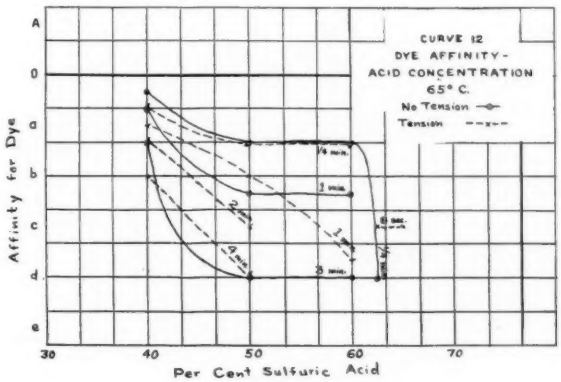
have untwisted, swollen and become cylindrical in shape, resembling cotton mercerized with sodium hydroxide very much. With polarized light they also give brilliant color effects, more so than untreated cotton, but with less intensity than caustic mercerized cotton. The samples treated similarly but without tension do not show the swelling and untwisting to the same degree. Those which have become stiff and "parchmentized" also show the swelling and untwisting, but the surfaces are very rough, showing that the acid has attacked the fiber. There was little evidence of any "cementing" having taken place between the fibers.

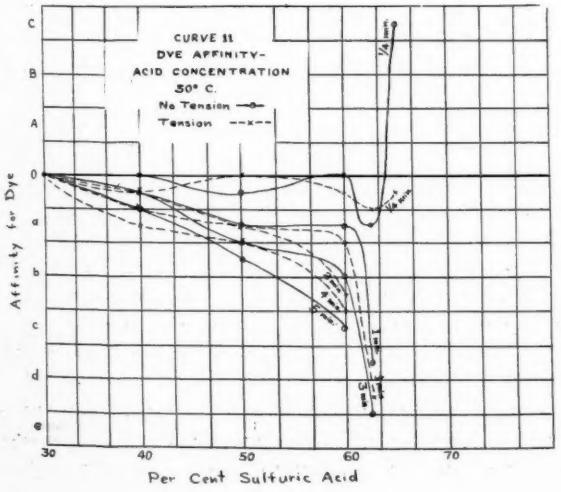
Theoretical Discussion

Sulphuric acid of 60 to 70% concentration seems to have a very specific action upon cotton. A maximum activity is obtained with 62.5% acid. Whether the reason for this specific action is chemical or physical was not determined. It was noted that the cotton absorbed a large amount of acid from solutions of 60 to 70% concentration. This was evidenced by the rapid change in concentration and the difficulty of washing all the acid out of the treated yarn. With concentrations below 60% no such absorption was noted. Swelling is also much more noticeable at these concentrations.

It is possible that the increase in strength up to concentrations of 62.5% acid in the bath is due to the swelling and increase in surface area of the fibers, which increases the surface-to-surface coefficient of friction, thus increasing the tensile strength. At higher concentrations chemical action between the fiber and acid probably begins, weakening the individual fibers by a degradation of the cellulose molecule and thus weakening the tensile strength of the entire thread.

The increased strength of the "parchmentized" product may be due either to the increased friction between the roughed surfaces of the fibers as they are pulled apart or to a "cementing" together of the individual fibers. In the latter case the fibers after swelling probably form a gelatinous coating of "hydrocellulose" on their surface; when the yarn is then removed from the acid and washed in water this is probably coagulated, adjacent fibers coagulating together at the same time and acting as if cemented or glued together.





Conclusions

1. Concentrations of sulphuric acid below 60% cannot be used for obtaining an improved luster on cotton yarn. A mercerized product may be obtained by using concentrations of 60 to 65% sulphuric acid if tension is applied during immersion in the acid; no improvement results if no tension is applied. The acid concentration should also be kept constant to obtain uniform results. Increasing concentrations above 65% show less mercerization than the above; concentrations above 70% immediately attack and dissolve the cotton.

TABLE VIII—65% SULPHURIC ACID

No.	Temp. (° C.)	Stretch	Time of Immersion	Affinity for Dye	Regain (P.C.)	Strength of Treated (Oz.)	Strength of Untreated (Oz.)	Change in Strength (Oz.)	Extens. of Treated (P.C.)	Extens. of Untreated (P.C.)	Change in Extens. (P.C.)	Shrinkage ±0.6% (P.C.)	Change in Shrinkage (P.C.)
A 1a	3–4	—	8 sec.	A—	7.7	22.4	19.2	+ 3.2	8.8	6.5	+ 2.3	1.3	0
1b	3–4	—	¼ min.	D—	7.7	22.8	19.2	+ 3.6	9.8	6.5	+ 3.3	2.8	+ 0.2
1c	3–4	—	1 min.	H	7.7	12.0	19.2	— 7.2	20.2	6.5	+13.7	31.0	+28.4
1d	3–4	—	2 min.	Disintegrated		...	...	...	...	...	...	...	...
2a	3–4	+	8 sec.	B—	7.7	21.8	19.1	+ 2.7	8.4	6.9	+ 1.5	Ext.1.2*	— 2.7
2b	3–4	+	¼ min.	C+	7.7	22.4	19.1	+ 3.3	8.2	6.9	+ 1.3	0	— 1.5
2c	3–4	+	1 min.	G	7.7	22.8	19.1	+ 3.7	5.1	6.9	— 1.8	Ext.0.9*	— 2.4
2d	3–4	+	2 min.	H—	7.7	20.6	19.1	+ 1.5	4.3	6.9	— 2.6	Ext.1.5*	— 3.0
B 1a	20	—	8 sec.	D—	5.5	21.6	18.1	+ 3.5	9.3	5.8	+ 3.5	2.8	+ 0.2
1b	20	—	¼ min.	F—	5.5	22.6	18.1	+ 0.6	11.2	5.8	+ 5.4	11.7	+ 9.1
1c	20	—	1 min.	Disintegrated		...	...	...	...	...	...	...	...
1d	20	—	3 min.	Disintegrated		...	...	...	...	...	...	...	...
2a	20	+	8 sec.	A+	5.5	21.3	18.0	+ 3.3	7.1	6.2	+ 0.9	1.8	0
2b	20	+	¼ min.	C+	5.5	22.6	18.0	+ 4.6	6.7	6.2	+ 0.5	1.6	0
2c	20	+	1 min.	E+	4.5	22.4	17.5	+ 4.9	3.7	5.8	— 2.1	Ext.2.7*	— 4.2
2d	20	+	2 min.	E	4.5	23.0	17.5	+ 5.5	3.7	5.8	— 2.1	Ext.0.8*	— 2.3
C 1a	50	—	8 sec.	B	6.4	19.1	18.6	+ 0.5	8.9	6.1	+ 2.8	3.5	+ 0.9
1b	50	—	¼ min.	C	6.4	9.3	18.6	— 9.3	6.9	6.1	+ 0.8	15.0	+12.4
1c	50	—	1 min.	Disintegrated		...	...	...	...	...	...	...	...
1d	50	—	3 min.	Disintegrated		...	...	...	...	...	...	...	...
2a	50	+	8 sec.	A+	6.4	18.3	18.5	— 0.2	6.5	6.5	0	1.3	— 0.2
2b	50	+	¼ min.	C—	6.4	16.9	18.5	— 1.6	4.9	6.5	1.6	0.5	— 1.0
2c	50	+	1 min.	Disintegrated		...	...	...	...	...	...	...	...
2d	50	+	2 min.	Disintegrated		...	...	...	...	...	...	...	...
D 1a	65	—	8 sec.	c+	6.4	9.9	18.6	— 8.7	6.1	6.1	0	2.5	0
1b	65	—	¼ min.	Too weak to determine values				...	...	...	...	...	...
1c	65	—	1 min.	Disintegrated		...	...	...	...	...	...	...	...
1d	65	—	3 min.	Disintegrated		...	...	...	...	...	...	...	...
2a	65	+	8 sec.	c	6.4	10.8	18.5	— 7.7	4.6	6.5	— 1.9	1.2	— 0.3
2b	65	+	¼ min.	Completely disintegrated				...	...	...	...	...	...
2c	65	+	1 min.	Completely disintegrated				...	...	...	...	...	...
2d	65	+	2 min.	Completely disintegrated				...	...	...	...	...	...

*An extension in length instead of a shrinkage occurs in these cases.

LUSTER

A-1. a, no change; b, decreased luster; c, no luster and harsh. All tinged slightly yellow.

A-2. a and b, slightly increased luster; c and d show increasing stiffness and parchmentizing. All tinged slightly yellow.

B-1. Luster decreased.

B-2. a and b, luster improved; c and d have some luster, but are stiff like straw, or "parchmentized."

C-1. a, no change; little luster on b, also swollen.

C-2. Slight improvement; b best.

D-1 and D-2. No change.

---

2. The time of immersion in the acid should be from ¼ to 2 minutes. Longer periods give a harsh feeling or parchmentized-like product.

3. Temperatures of 0 to 30° C. can be used to secure the improved luster. Temperatures higher than this tend to weaken the cotton and decrease its luster. It is possible that temperatures of —10 to —20° C. would give even better results. The maximum effect in this work was obtained at 0 to 20° C.

4. The tensile strength and extensibility show a marked increase only with concentrations of 62.5 to 65.0% sulphuric acid. Concentrations below this affect the strength only slightly at ordinary temperatures; at higher concentrations the cotton is weakened very much.

5. Concentrations of 60 to 70% sulphuric acid will increase the affinity of the cotton for direct dyes. It should be possible to increase both the affinity for dyes and the strength by using temperatures below 0° C. with these concentrations of acid.

6. Increase of strength and dye affinity result either with or without the use of tension. However, the use of tension gives a slightly stronger product than

TABLE IX—70% SULPHURIC ACID

No.	Temp. (° C.)	Stretch	Time of Immersion	Affinity for Dye	Regain (P.C.)	Strength of Treated (Oz.)	Strength of Untreated (Oz.)	Change in Strength (Oz.)	Extens. of Treated (P.C.)	Extens. of Untreated (P.C.)	Change in Extens. (P.C.)	Shrinkage ±0.6% (P.C.)	Change in Shrinkage (P.C.)
A 1a	0–5	—	8 sec.	E	7.5	12.0	19.1	— 7.1	6.5	6.5	0	5.1	+ 2.5
1b	0–5	—	¼ min.	(†)	...	...	...	...	...	...		Approx. 25	...
1c	0–5	—	1 min.	Disintegrated, swelling first				...	...	...	...	...	...
1d	0–5	—	3 min.	Disintegrated, swelling first				...	...	...	...	...	...
2a	0–5	+	8 sec.	D+	4.5	19.3	17.5	+ 1.8	5.1	5.8	— 0.7	Ext.1.2*	— 2.7
2b	0–5	+	¼ min.	G	4.5	20.2	17.5	+ 2.7	4.2	5.8	— 1.6	Ext.1.6*	— 3.1
2c	0–5	+	1 min.	H	4.5	17.7	17.5	+ 0.2	3.0	5.8	— 2.8	Ext.2.3*	— 3.8
2d	0–5	+	2 min.	H	4.5	12.2	17.5	— 5.3	2.2	5.8	— 3.6	Ext.1.0*	— 2.5
B 1a	20	—	8 sec.	(†)	...	...	...	...	...	...		Approx. 30	...
1b	20	—	¼ min.	(†)	...	...	...	...	...	...		Approx. 75	...
1c	20	—	1 min.	Disintegrated, swelling first				— 0.7	5.4	6.9	— 1.5	Ext.0.3*	— 1.8
1d	20	—	3 min.	Disintegrated, swelling first				— 7.7	2.3	6.2	— 3.9	Ext.1.1*	— 2.6
2a	20	+	8 sec.	E—	7.5	18.3	19.0	...	...	...	...	...	...
2b	20	+	¼ min.	I	5.5	10.3	18.0	...	...	...	...	...	...
2c	20	+	1 min.	Disintegrated, swelling first				...	...	...	...	...	...
2d	20	+	2 min.	Disintegrated, swelling first				...	...	...	...	...	...

*An extension in length instead of a shrinkage occurs in these cases.

†Note: A-1b, stiff, lusterless mass, threads stuck together; B-1a, shrinks to lusterless threads, much stiffer than A-1b; B-1b, shrinks to a lusterless mass, more stiff than B-1a; B-2a, soften than B-2b but no luster; B-2b, without luster.

All of the above samples dyed very unevenly.

if no tension were used. The affinity for dye is independent of the use of tension in the mercerization.

7. With concentrations of 60 to 70% sulphuric acid and without the use of tension, a shrinkage in length is obtained, which increases very rapidly with increasing time of immersion. If treated with tension an increase in length results; however, this increase amounts at the greatest to only about 4%, compared to shrinkages obtained without the use of tension of 30% and greater.

8. Sulphuric acid of 62.5% concentration shows a maximum activity with cotton, affecting all its properties more at this concentration than at any other.

(The End.)

SUGGESTS CONTROL OF CALENDERING FOR GREATER EFFICIENCY

In Paper Read Before Textile Engineers at Greenville Meeting

DECLARING that calendering has become a more important process in the manufacture of cotton fabrics than ever before, James A. Campbell, production manager of the Rockland Finishing Company, told a body of textile engineers recently that the most efficient operation of calendering machinery may be attained by more scientific control. He said, moreover, that calendering as a process has lately come in for considerable study, "where before it was much neglected and done in a more or less hit-or-miss fashion, depending on the ideas of the men in charge of the plant."

The substance of his paper, which was read at the recent meeting in Greenville, S. C., of the Textile Division of the American Society of Mechanical Engineers, was as follows:

"The outstanding improvements in calendering have been mostly in actual machine operation, and next in improving the design, strength and weight, the methods of driving the machines. There has been, however, no change in the underlying principles of the machines themselves.

"As the basis of the best operation of any calendar is derived from careful time studies, the progressive finishing plant, therefore, has to make numerous checks of all the elements in calendering, so that all unnecessary operations are eliminated and the machine will be handled in the best possible manner to secure the maximum production with the lowest cost.

"Accurate time studies have brought out, of late, facts which were only more or less recognized a number of years ago. It is my opinion that:

"1. There will probably be no radical changes in the underlying principles on which the machines operate.

"2. There will be a more general knowledge of the facts of calendering.

"3. Operation will be based more and more upon scientific facts and definite control of operation.

"4. Quantity and quality will increase through better control of: (a) Temperature, (b) pressure, (c) conditions in which the goods arrive at the calender, (d) better methods of handling, (e) further experiments should be carried on to run two rolls of cloth at one time on chase calenders, (f) more consideration should be given to mechanical handling of cloth rolls."

Early Documentary References on the Use of Dyestuffs in America*

The Myth of the "Sad-Color" Puritan—Governor Winthrop as Dye Chemist—When Our Ancestors Dyed Homespun—Some Early Vegetable Colors—Dyeing Troubles of a Century Ago—Patriotism and a Home Dye Industry in 1812—Formulas from an Old Notebook

By DR. C. A. BROWNE

Chief, Chemical and Technical Research, Bureau of Chemistry and Soils, Washington, D. C.

THE members of this division who have read Sir Maurice Low's interesting volume upon "The American People" will perhaps recall a chapter which is entitled "The Puritan Hatred of Color a Myth." After having demolished the story of the blue laws and a goodly number of other fictions which have been fastened upon the Puritans. Sir Maurice shows that the reputed hatred of these people for colored fabrics was also a legend. The tradition seems to have had its origin in a mistaken idea as to the meaning of the expression "sad colors" which was used generally by the Puritans and all English-speaking people three centuries ago. The word *sad* when employed in this connection did not mean sombre or dark, as wrongly interpreted, but any color of a quiet or subdued tone.

A mere reference to the documents and literature of the period is sufficient to establish this point, as, for example, the following enumeration of sad colors in an English list of dyes for the year 1638: "Sadd-colours, the following: liver colour, De Boys, tawney, russet, purple, French green, ginger-lyne, deere colour, orange colour," which is enough to prove that even in the selection of the so-called "sad colors" the Puritans did not limit themselves to blacks or grays.

But the Puritans who first settled the Bay District of New England employed also colors of the more vivid type which are not included in the "sad" list, such as red, yellow and blue. They loved a variety of shades. John Winthrop, the first governor of Massachusetts, gives the following directions to his son in the purchase of dress goods: "For the stuff for the gowns you may buy it of some olive colour, or such like. Either let there be several colours or else the velvet for the capes of several colours."[1] And again in a later list of necessaries which he desired his son to bring from England, Winthrop enumerates: "Three hundred sheep skins and lamb skins, with the wool on, dyed red; and the coarsest woolen cloth and of sad colors and some red."[2]

The younger Winthrop to whom this letter was written was a man of considerable chemical attainments, and for the prominent part that he took in establishing such industries as those of iron, salt, saltpeter, pitch, tar, potash and various others, which can be named, deserves to be called the pioneer chemist of the English Colonies. He made the first importations of chemical to New England, and he possessed probably the first chemical library within the present borders of the United States. Many of his chemical books can still be seen in the Society Library of New York City. His original manuscripts and papers, which are preserved in the library of the Massachusetts Historical Society at Boston in the priceless collection of Winthrop papers, contain many references to chemistry and chemical industries. Among these documents we find a number which relate to dyestuffs and to dyeing. Winthrop was interested at one time in the possible introduction of Indigo culture in New England, and there is still preserved a memorandum of about the year 1650 in his handwriting upon the manufacture of Indigo. This document, which is one of the oldest original papers relating to American chemical industries, reads as follows:

"Indigo. The Receipt for Making of Indigo.—One or two houres after the herb is cutt, lay it in a fatt, presse it downe hard with a beame over cross barres that aire may come to it till it worke & raise the barrs, let it lye 24 houres, then fill the fatt halfe full of water till the weede rott in the water, usually in 24 houres, then fill the fatt full. So lett it stand untill it come to a coulor within 3 daies tyme the weede unrotted take out lett the rest stand 24 houres more, then stirre it that it may all runne out into an other fatt; then beate it & poure it in & out with bucketts & that incessantly, till it come to one perfect coulor, lett it then settle, make then a tap to draw forth all the thin water, then take up the bottom remaining into baggs that will hold 5 pound weight, made of strong canvasse with an hoope on the top, & then a stick acrosse, by which hang it in a house & save the droppings, which will make a good Coulor (so the first drawne water a reasonable coulor) in an houres

*Paper presented before the Dye Division of the American Chemical Society at its meeting held in Swampscott, Mass.

[1] Life and Letters of John Winthrop, Vol. I, p. 251.

[2] Ibid., Vol. II, p. 60

tyme the water will all dropp out of the bagge, then take the remaining Indico into boxes, in which lay the Indico some 3 fingers thick, which set in the sunne & let them candy (else in an oven or stone to dry temperately not in hast) then whilest it is drying slice it with a knife."[3]

Winthrop's interest in dyestuffs was no doubt partly the result of his friendship with Abraham Keffler, of Leyden, Holland, one of the famous names in the history of dyeing. We have as evidence of this friendship an interesting annotation in a small duodecimo volume, bound in leather, which once belonged to Winthrop's library. The book contains two treatises by that mythical writer of alchemistic tradition, Basil Valentine, and its blank pages bear numerous notes in Winthrop's handwriting. The most interesting of these annotations is the one which was penned by Winthrop upon the first fly-leaf.

"This was once the booke of that famous philosopher and naturalist Cornelius Drebbel, who usually carried it with him in his pockett and after his death was given me by his sonne-in-law, Mr. Abram Keffler. —John Winthrop."

In the library of the Massachusetts Historical Society there is preserved a letter to Winthrop by the Abraham Keffler above mentioned. It is dated June 12, 1639, and reads in part as follows:

"I know you desirous to heare of my prosseeding in Alchimie; all my prosseeding therin is lost, by reason of my long sickness, sso that I am beeginning agayne . . . I now onely follow dieing of scarrllett, in which I have so much to doe that I can follow nothing elles." This old letter of Keffler to his friend Winthrop, with its reference to the "dieing of scarrlett," recalls one of the most interesting discoveries ever made in the technology. The account of it, as given in Beckmann's "History of Inventions," is as follows:

"The well-known Cornelius Drebbel, who was born at Alkamaar, and died at London in 1634, having placed in his window an extract of cochineal, made with boiling water, for the purpose of filling a thermometer, some aqua regia dropped into it from a phial, broken by accident, which stood above it, and converted the purple dye into a most beautiful dark red. After some conjectures and experiments, he discovered that the tin by which the window frame was divided into squares had been dissolved by the aqua-regia, and was the cause of this change. He communicated his observation to Kuffelar, an ingenious dyer at Leyden, who was afterwards his son-in-law. The latter brought the discovery to perfection, and employed it some years alone in his dyehouse, which gave rise to the name of Kuffelar's colour. Becher calls him Kuffler, but Kunkel, in a passage which I cannot again find, makes his name Kuster, and says that he was a German. In the course of a little time the secret became known to an anabaptist called Gulich, and also to another person of the name of Van der Vecht, who taught it to the brothers Gobelins in France."

The use of salts of tin in dyeing, one of the most epoch-making discoveries in the history of the art, dates from the early work of Winthrop's friend Abraham Keffler.

When Our Ancestors Dyed Homespun

The first use of dyes in New England, as well as in the other North American colonies, related to the coloring of homespun. It was largely a domestic operation that was carried out in every home, the Indigo pot and dye kettle being a part of the ordinary household utensils. Somewhat later, when the housewife wished to relieve herself of some of her innumerable occupations, as well as of the pollution of the home atmosphere by stenches from the dye pots, the homespun was sent to someone who made a specialty of the art of dyeing. We can form some idea of the equipment of one of these early domestic dye establishments from the following items in the inventory of the estate of John Cornish, of Boston, which was taken in March 1695/6:

82 lbs. of copperas	0/13/9
34 lbs. of galls	2/11/0
70 lbs. of redwood	3/10/0
60 lbs. of potash	1/10/0
35 lbs. of madder	1/15/0
230 lbs. of fustick	2/13/0
1 jack, 2 spits, 1 pair andirons, fire shovel, tongs and 2 trammels	3/ 0/0
2 pots, 2 kettles and 1 skillet	2/ 0/0
barrels, tubs and lumber	0/ 6/0
2 pair scales	0/16/0

The four dyes enumerated in the above list, galls, redwood, madder and fustic, were all imported, although madder was cultivated to a limited extent in some of the North American colonies. Small domestic establishments, similar to the one of John Cornish, existed in many parts of the country until after 1800. Chastellux, in his "Travels in North America," in the years 1780-81-82, mentions in the course of a visit to Hartford: "I went into a house where they were preparing and dying the cloth. The cloth is made by the people of the country and is sent to these little manufactories where it is dressed, pressed and dyed for two shillings, lawful money, per yard." It was only when homespun ceased to be made that the little domestic dyehouse, forming in many cases a part of the owner's home, ceased to exist.

The early settlers obtained much information from the native Indians regarding the tinctorial properties of various indigenous plants. The Indians employed

[3] Massachusetts Historical Collections. Series 4, Vol. VI, p. 82.

an infusion of the bark of the chestnut-oak for imparting a red color, and of the three-leaved Hellebore (*Helleborus trifolius*) for imparting a yellow color, to tanned skins. The Indians used extracts of other native plants, such as *Galium tinctorium*, puccoon, pokeberries, etc., for coloring feathers, porcupine quills, bark and other pieces of fancy work and this knowledge was put to a practical use by the first white immigrants in the dyeing of their homespun.

With very few exceptions the dyes used in the North American colonies or in the United States previous to 1890 were vegetable colors. Schoepf[4] mentions a method of mineral dyeing that was practised about the time of the Revolution, which he describes as follows: "For each pound of the yarn to be dyed, a pound of the purest copperas is taken. The yarn is dipped first in a clear, warm lye, and then into the copperas solution, the dippings repeated six to eight times, but each time the yarn should be a little while hung up to air; in this way, it is said, a deep straw color is given the yarn." Prussian Blue, Copper Blue, Chrome Yellow and other mineral colors were not used for dyeing until some time after the Revolution.

It will be interesting to enumerate a few of the vegetable colors which were used for dyeing in the early history of our domestic tinctorial art.

Some Early Vegetable Colors

Black

Infusions of the leaves and berries of the gall-berry or ink-berry (*Ilex globra*), which grows plentifully on the shores of bays, creeks and ponds along the Atlantic seaboard, were used for giving a black color to hats, yarn and similar goods. The juice of baneberries (*Actaea spicata*), when boiled with alum, also afforded a black dye. The capsules and bark of many species of oak were also used as a basis for producing black, copperas or alum being used as mordants, or setting ingredients, to employ the old expression. Infusions of the bark of other trees, such as the red maple (*Acer rubrum*), were also used. The barks were ground, boiled with water, strained and then treated with copperas or alum before putting in the fabric to be dyed. Where the imported logwood was available, this was the most generally used dye for coloring black. Peter Kalm, the Swedish naturalist, who traveled through the American colonies in 1753-1761, reported that the people spun and wove a great part of their every-day's apparel, and dyed them in their own houses. The method which he observed the colonists to use for dyeing black is described as follows: "To get a black color, the leaves of the common field sorrel (*Rumex acetosella*) are boiled with the stuff to be dyed, which is then dried and boiled again with logwood and copperas; the black color thus produced is said to be very durable."[5]

Blue

Indigo was the dyestuff which was used most generally in all the North American colonies for coloring blue. All the Indigo used in the colonies was imported until about 1742, when its cultivation was introduced in South Carolina from the West Indies by Miss Eliza Lucas. Its production in the colonies was encouraged by a bounty of sixpence per pound, passed by Parliament in 1748, upon all the Indigo raised in the British-American colonies and imported directly into Great Britain. In 1754 the export of Indigo from Carolina amounted to 216,924 pounds, and this amount had increased to 1,107,660 pounds in the year preceding the American Revolution. The production of Indigo in the South constituted one of the most important chemical industries of the United States until the War of 1812, when the cultivation of cotton began to supplant the other crop. The method of manufacturing Indigo in the Carolinas, as observed by Schoepf[6] just after the Revolution, was as follows:

"In cutting and gathering, the herb must be gingerly dealt with, so that the bluish farina, which covers the leaves and is said to add much to the richness and beauty of the color, may not be rubbed off; nor should the plant be bruised, for if so its heavy juices spoil the delicacy of the color, which must be got merely by fermentation of the unmangled plants steeped in water. To this end, they are carefully placed in a vessel, the 'steeper,' 10 to 15 feet long and 4 feet deep, the plants lying 12 to 15 inches deep, over which water is poured; according to the state of the weather; after 12 to 18 hours the plants begin to warm of themselves, swelling and fermenting; the time of the greatest and most complete fermentation must be carefully observed; the method in use is to place a thin stick over the mass which rises as it rises, but should the mass fall below the point where the staff is propped at the sides of the vessel, it is then time to let off the water into another vessel called the 'beater.'

"In this vessel the water, charged with the color-particles thrown off in the steeper by fermentation, is by a peculiar process beaten until it begins to foam and rise over the sides, which happens according to the warmth of the weather in 25 to 30 minutes, more or less. To check a too vehement overflow of the material a little oil is poured on, which has an immediate quieting effect. This beating of the water furthers the association of the color-particles contained

[4] Travels in the Confederation (1783-84). From the German of Johann David Schoepf, Philadelphia, 1911; translated by Alfred J. Morrison. Vol. I, p. 176.

[5] Travels into North America (1753-61), by Peter Kalm. (Translation by John Reinhold Forster, Londton, 1772. Vol. I, p. 275.)

[6] Travels in the Confedation (1783-84). From the German of Johann David Schoepf; translation by Alfred J. Morrison. Philadelphia, 1911. Vol. II, pp. 157-159.

in a dissolved state; so as not to miss the point of time when this begins to take place, a few drops of the beaten water are at intervals taken on the finger nail, on a tin plate, or in a glass, and so soon as a blue shimmer is observed, or blueish particles show themselves, this process almost must be discontinued. There is then, with a gentle stirring a proportionate quantity of lime-water poured on, which brings about the precipitation of the Indigo; when this is fallen to the consistency of a thick broth, the water standing above (now clear) is drawn off, and the sediment put into bags and hung up until the moisture has largely come away.

"Finally this mass is taken out of the bags, kneaded on boards and wooden spades, divided into little cakes, and thoroughly dried, regard being had to the morning and evening sun. The preparation of Indigo, which here and there is carried on with certain variations, is on the whole a chemical process requiring the most careful and exact attention in all its parts, the essential depending always on the right use of the proper moment, at which this or that should be done: The quality of the Indigo is as much due to the exactness of its preparation as to the nature of the plant, of the soil, and of the weather. Hence Indigo planters have not always equally good fortune, and often lose by the unskilfulness, malice, or carelessness of their headmen and workmen, much or the whole of a crop. The headmen in this sort of work are commonly negroes, and if they thoroughly understand the management of the Indigo, a great value is set upon them, and they often fetch two or three times as much as they would ordinarily."

The colonial process of dyeing with Indigo and the one employed generally in the United States until after the War of 1812 was to allow the finely pulverized dye to remain in contact with fermenting urine or swill in a pot or tub to which lime or wood ashes was added to maintain a slightly alkaline reaction. Bran was also frequently added to the fermenting mixture. The Indigo pot was usually set in a warm place near the hearth stone and the frequent references to the malodorous atmosphere which emanated from this utensil when it was in full operation are probably not overexaggerated. After the Indigo was reduced to the soluble leuco-form the fabric or yarn was worked in the liquor and then hung up in the air until the blue color was reformed by oxidation. The dyed material was then washed and dried. The collection of urine, or chamber-lye, for use as a detergent, mordant and reducing agent in the dye establishments of the United States continued until the Civil War. It was employed in the preparation of other dyes besides Indigo.

Red

The use of puccoon or blood root (*Sanguinaria canadensis*), sometimes called bastard turmeric, which the colonists learned to employ as a red dye from the Indians, has already been mentioned. The roots of crosswort and of several other native species of Galium were used for imparting a red color to wool. The fruit of the sumac (*Rhus glabra*) was also employed as a red dye. Madder (*Rubia tinctorum*), however, was the principal material which was used for dyeing fabrics red until the introduction of the synthetic colors. The madder was largely imported, although repeated attempts were made to introduce its cultivation. The author of the famous early treatise, "American Husbandry," laments the fact that so much madder was imported when there were such extensive tracts of land in the colonies available for its cultivation. "No article of American husbandry," he remarks, "would prove a more valuable staple." But the farmers who experimented with madder soon abandoned it for other more profitable crops. Of other imported dyes which were used for coloring red may be mentioned brazilwood, sanderswood, camwood, and lac.

Yellow

Probably more plants and plant products were used for the production of yellow in colonial times than for any other color. The roots of the nettle, barberry, yellowroot (*Xanthorrhiza apiifolia*) and other plants, the bark of the apple, plum, hickory and other trees, leaves of the birch, sweet leaf (*Symplocos tinctoria*) and numerous other shrubs, flowers of the marigold, sunflower, safflower and other plants, and numberless common weeds, such as smartweed, knapweed, jewel weed, etc., were variously used for dyeing yellow. Of the imported vegetable dyes tumeric, saffron, quercitron and fustic were the ones most generally used for producing yellows. Attempts were made to introduce a saffron industry in the North American colonies, but the results were not wholly successful. The author of the treatise on "American Husbandry" remarks with regard to the cultivation of saffron in New Jersey:

"In the most southern parts of the province, saffron is commonly planted; but the drug produced is not reckoned so good as the product of England. This is not to be attributed to either soil or climate, for both suit it in an extraordinary degree; but they are not careful enough in the culture nor in the manufacture of the commodity after it is produced; they do not weed the crops with that assiduous care which the planters of Cambridgeshire and Flanders exert, and which seems to be essential to the success; nor are they equally attentive to curing, drying, and caking of it."

Orange

Among the substances used in colonial times for producing orange, Peter Kalm mentions the bark of

the sassafras.[7] He writes: "The bark of the sassafras is used by the women in dyeing worsted a fine lasting orange color, which does not fade in the sun. They use urine instead of alum in dyeing and boil the dye in a brass boiler, because in an iron vessel it does not yield so fine a color." Of the imported substances for dyeing orange, annatto was probably the dye most generally used.

Brown

The favorite color of homespun, when worn for every-day use, was brown, and for producing this shade a large number of common vegetable dyes were employed by the early colonists, such as the bark of the butternut, the walnut, the white oak, the alder, the maple and other trees. Of the imported dyes, cutch was probably the one most used for coloring brown.

Scarlet and Purple

The juice of pokeberries (*Phytolacca decandra*) was used by the early colonists for producing a variety of shades. Peter Kalm mentions the high purple color of this juice which he states "is as fine as any in the world; and it is a pity that no method is as yet found out, of making this color last on woolen or linen cloth, for it fades very soon."[8] Pokeberry juice, treated with alum, was used for coloring crimson and, treated with lime, for producing yellow. Of the imported dyes cochineal was chiefly used for the production of scarlet.

The list of early dyes which have been named could be greatly extended, but enough has been mentioned to show that the settlers of North America in colonial and later times had at their disposal a large assortment of domestic and foreign dyestuffs which, either singly or in mixture, enabled them to produce almost any shade of color that was desired.

Dyeing Troubles Before 1812

As compared with European standards the art of dyeing in America was in a very backward state until after the War of 1812. The prevailing defect of American goods was the lack of fastness in their colors. The proper manipulation of mordants was not understood and the distribution of the dyes in the fabric was not always uniform. Undyed edges were the distinguishing mark by which American goods were recognized.

With the great stimulus which was given to all domestic industries during the War of 1812 there was an overzealousness on the part of American textile manufacturers to imitate all the varied colors of the best foreign dyed fabrics. An advertisement of the Housatonick Factory at Pittsfield for July, 1816, offered to dye homespun cloth and to take in payment wool, flax, wood, soap and all kinds of country produce, for all colors except Indigo Blue for which part cash was expected.[9] Ready money was lacking at that time and dyeing, like every other business, had to be conducted upon a system of barter.

The willingness of manufacturers to dye all colors resulted in many disappointments. A local historian of this period states: "The earlier American manufacturers had not learned the wisdom of division of labor and concentration of effort. Each little factory set itself to satisfy the varied demands of the universal market; and the advertisements of the first Pittsfield mills read like descriptions of diverse spectra, or an enumeration of the colors of the rainbow imparted to every known fabric of wool. Thus, before the manufacturer had discovered the source of his failure in one class of goods and devised or learned a remedy, he was called to another, in which he encountered new and mysterious troubles; and so on in an endless circle of tribulations."[10]

Many of the failures of efforts to reproduce the colors of European dyed goods during this period were attributed to sabotage on the part of workmen who had been bribed by foreign manufacturers. Accusations of this kind were common, however, in all industries, and must be duly discounted as reflections of the disturbed temper which comes from every international conflict.

Henry B. Fearon, an English observer of the period following the War of 1812, makes the following rather contemptuous remarks[11] for the benefit of British readers about the low state of the dyeing business in the city of New York:

"Mr. W. and Son, who are dyers, would find some difficulty in stepping from the practice of their large concern . . . to an American dyehouse; which, from the non-existence of manufacturers in this country, must be essentially different from an English one. The articles usually done here are old clothes and spoiled goods. A dyer's business in this city will best accord with an English scourer. . . . The price for dyeing black woolen is 3 s. per yard 6/4 wide; of brown ditto, 3 s.; red, 2 s. 6 d.; yellow, 2 s. 6 d.; scarlet, 20 s. a pound. There is no silk dyed in the skein, nor are there any silk weavers in the United States. Fast blue is not done. Redyeing old silk is 6¼ d. per yard. English alum is from 33 s. 9 d. to 56 s. per cwt., to which is to be added a duty of 4 s. 6 d.; brazilletto, 140 s. to 160 s. per ton; cochineal, 24 s. 9 d. per pound, with a duty of 7½ per cent; logwood, 90 s. to 112 s. 6 d. per ton. The business is of necessity limited. It it moderately good and would not require a capital of more than from 200£ to 500£. A few journeymen are employed. They earn 2£ 5 s. per week."

(*To be concluded in the next issue*)

[7] Travels into North America (1753-61), by Peter Kalm. (Translation by John Reinhold Forster, London, 1772.) Vol. I, p. 115

[8] Ibid., Vol. I, p. 153

[9] Smith's "History of Pittsfield, Massachusetts," Vol. II, p. 471.

[10] Smith's "History of Pittsfield, Massachusetts," Vol. II, p. 489.

[11] "Sketches of America," by H. B. Fearon, London, 1818, p. 31.

Our Dye Production in 1927 Exceeded All Records

Tariff Commission's Latest Census Reports Many New Colors Developed—Vat Dye Output Also Reached New Mark—International Situation Outlined

EVERY year the domestic dyestuff industry outstrips its previous records of production so that a new total no longer creates the excitement that it did a decade ago. But there is far more than total poundage to think about in the eleventh annual Census of Dyes and Other Synthetic Organic Chemicals issued last week by the United States Tariff Commission. The report shows that the domestic industry made marked progress in more directions than merely that of record production.

As intimated, the output of more than 95,000,000 pounds of dyes for 1927 exceeds that of any previous year. This total, incidentally, is a gain of more than thirty million pounds over that established five years ago in 1922.

American dyes supply approximately 94 per cent of this country's requirements, and in 1927 there was in addition an exportable surplus amounting to over 26,000,000 pounds, as compared with 1913 when only 13 per cent of the dyes consumed were produced in this country, chiefly from imported intermediates. The domestic industry has not been content merely to follow in the footsteps of the older dye-producing nations, but has developed many new and important dyes.

In 1927 considerable progress was made in the manufacture of fast and specialty dyes, including those for dyeing rayon and mixed fabrics. The production of vat dyes established a new record in that year, with a total of over 5,960,000 pounds, as compared with 4,030,000 pounds in 1926. This class of dyes was not made in the United States prior to the World War. As a result of the insistent demand of the public for fast-dyed fabrics the consumption of vat dyes has been increasing each year. These dyes are used largely on cotton goods and yield shades of exceptional fastness to sunlight and washing. The variety and beauty of shades as well as the fastness of vat dyes have been responsible for the increase in their use.

The production of coal-tar dyes by 55 firms in 1927 was 95,167,905 pounds, an increase of 8 per cent over the previous year's production. Sales in 1927 totaled 98,339,204 pounds, valued at $38,532,795, at a weighted average of 39 cents per pound, or 3 cents per pound less than the corresponding average price during 1926. The average price in 1917 was $1.26 per pound. Competition between manufacturers has been quite severe and 38 firms have gone out of business since 1919.

Imports of dyes during 1927 were 4,233,046 pounds, with an invoice value of $3,413,886 representing a decrease of 10 per cent by quantity, and 16.5 per cent by value from that of 1926.

Exports of dyes in 1927 showed an increase in quantity but a decline of 8 per cent by value from that of 1926.

International Dye Trade

Severe competition among the dye-producing nations of the world continued throughout 1927, as it has for several years past. In time the cartel arrangements entered into by the dye industries of foreign countries will doubtless check this excessive, international competition. These agreements have as their main objects the division of the world's markets, the limiting of production, and the stabilization of prices.

A number of agreements were negotiated in 1927 between important foreign dye manufacturing groups. The most important of these was the Franco-German dye agreement which was entered into by groups representing virtually the entire dye industries of both countries. In these negotiations the governments of both countries conferred constantly with the two groups.

By the terms of the agreement competition between France and Germany in dyes is eliminated. It was stated that the agreement was not directed against other countries, but that in time it would be extended to them. It is expected that France will undertake the manufacture of new dyes, receiving technical aid from Germany. The agreement assigns an export quota to France exceeding by 1,500 tons the exports during the last year.

The quantity of dyes exported from Germany in 1927 was less than half the quantity of exports in 1913, but the value shows an increase of 7 per cent in 1927 over the pre-war year. The decrease in volume and increase in value of dye exports are due to heavy shipments of high-priced dyes and to a loss in exports of low-priced bulk colors. Export statistics, taken alone, do not disclose the actual participation of Germany in international trade as she controls or has an interest in dye plants in Japan, Spain, Italy, the United States and Russia.

No announcement has been made of British producers entering into any international dye agreements and it is considered unlikely that any such agreements will be concluded. Early in 1928 the Finance Company of Great Britain and America, Ltd., was formed. The principal parties to this agreement were the Imperial Chemical Industries, Ltd., the largest dye and chemical company in Great Britain, and the Chase Securities Corporation in New York.

The three principal Swiss manufacturers have plants established in Germany, France, England, Italy,

Poland and the United States. These plants—owned and operated jointly—supply large quantities of dyes direct to consumers in their respective localities. About 90 per cent of the Swiss dye production is exported. Exports of all dyes in 1927 were greater than in 1926 but exports of Indigo continued to decline.

The dye industry of Italy now supplies the domestic needs of the cheaper bulk colors and has an exportable surplus.

Medicinals

A special feature of the 1927 Census of Dyes and Other Synthetic Organic Chemicals is a section devoted to the discussion of synthetic medicinals. These are of far-reaching importance to the health of the nation, and their manufacture in this country should be encouraged by all legitimate means. Without these products the cure of many diseases would be difficult and in some cases would be virtually impossible.

The prevention and cure of disease involves complex problems that no one branch of science is capable of solving. Fundamentally chemical in their nature, their solution nevertheless depends upon cooperation between chemists and other scientists. In reducing the ravages of disease, the role of the chemist is an important one. Advances of chemistry in the field of medicine in the past are indeed noteworthy; those in the future may be beyond the horizon of our present imagination.

ACCIDENT RATE LOWEST IN TEXTILE INDUSTRY

President of Cotton Association Quotes Figures for Safety Congress

THE position of the textile industry in relation to the accident rate was brought out by Russell T. Fisher, president of the National Association of Cotton Manufacturers, in an address before the National Safety Congress held recently in New York. The title of Mr. Fisher's address was "Safety in the Cotton Industry," and during the course of it he quoted facts and figures made public by the National Industrial Conference Board.

Of the seventeen major industries of the country, the textile industry had the fewest number of accidents causing death, with the exception of the tanning and packing industry, where the information was not complete. In the number of accidents causing temporary disability the textile industry is lowest; in accidents causing permanent disability next to the lowest, public utilities being the only industry with less.

The well-established fact that accidents causing temporary disability are the chief concern of the textile industry is further substantiated by this report, with 30.33 out of a total of 31.48 accidents in this class. In considering the total number of accidents the textile industry is considerably below any of the other industries, the chemical industry being the nearest, with 43.81 per cent accidents, compared to 31.48 per cent for the textile industry.

Accident Frequency, 1926

(Number of accidents per 1,000 workers per year)

Industry	Death	—Accidents Causing— Permanent Disability	Temporary Disability	Total Accidents
Mining	2.37	1.99	180.40	184.76
Woodworking	.75	3.13	144.97	148.85
Packers and tanners*	...	1.60	139.70	141.30
Construction	1.43	4.37	132.66	138.46
Quarries	2.04	2.67	132.02	136.73
Paper and pulp	.42	1.27	86.56	88.25
Not classified	.83	2.23	82.15	85.21
Metals	.44	2.79	81.86	85.09
Public utilities	1.40	.57	79.05	81.02
Rubber	.13	1.17	70.86	72.16
Petroleum	.57	2.23	67.36	70.16
Power press	.16	2.64	55.33	58.13
Food	.17	2.04	54.42	56.63
Automotive	16	2.72	53.73	56.61
Cement	1.11	1.58	53.58	56.27
Chemical	.61	1.72	41.48	43.81
Textile	.09	1.06	30.33	31.48
All industries	.54	2.22	73.73	76.49

*Report covers sixteen establishments.

Several of the insurance company's officials admit that there has been great progress in safety work in mills in the past several years. They feel that practically everything that can be done to safeguard machinery has been done, and that records show that accidents from mechanical causes are less frequent than in any other industry. They are now urging more educational work to cut down the non-mechanical accidents.

SWISS, TOO, USE OUR INTERMEDIATES

According to Vice-Consul Scott of Basle, large quantities of coal tar crudes and intermediates, such as toluidine and dimethylaniline are used by the Swiss dye industry. The largest imports of these materials come from Germany and some are also received from France, Austria, Great Britain and Belgium. The value of imports from the United States in 1926 was about 21 per cent of the total. The plants of the Basle dye concerns manufacture practically all of the dyes produced in Switzerland and their products constitute one of the principal Swiss export items. The value of the exports of aniline and other dyes in 1927 reached $13,810,100.

We do not buy from Switzerland as large a percentage of dyes as they buy in intermediates from us.

AMERICAN
DYESTUFF REPORTER

Devoted to the use and application of dyestuffs and the mechanical equipment incidental thereto.

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PROFESSOR OF CHEMISTRY AND DYEING, LOWELL TEXTILE SCHOOL
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STILL MORE DYES, FEWER DOLLARS

THE latest chapter in the story of American dyes is merely outlined in the report on the Census of Dyes for 1927, published elsewhere in this issue of the REPORTER. But even this bare outline contains enough real meat to send alert readers in the industry to the Census itself for the full details of the whole story.

Between the lines of the Tariff Commission's interesting report most readers will easily perceive one important fact about our dye industry: the industry moves ahead steadily year after year in several directions; it keeps advancing. In its control of 94 per cent of domestic needs, in its output of vat colors, in its gains in exporting, in its steady reduction of prices, there are indications that the dye industry of this country is constantly active, and largely in a beneficial way. Only in the vital matter of net profits is progress slow.

The record of our dye export trade for the year 1927 shows a gain of 200,000 pounds in quantity—small, perhaps, in comparison with other years, but it is a distinct gain. The value of these exports, however, dropped off 8 per cent below the value for the previous year. In 1926 the value declined 11 per cent from the previous year. That makes a 19 per cent decline in two years in the amount our dye manufacturers receive for the products they sell out of the country. Competition from European chemical trusts and some rather strong rivalry among the larger domestic firms are plainly responsible for this loss in sales value that our industry endures in the export field. A more united industry is probably the best solution to the problem of making money from exports.

The movement in this country for faster colors, however, will eventually lead to profits that may offset the loss in the valuation of our dye exports. In its increasing output of vat colors the domestic industry clearly shows that it is well prepared to supply the swelling demand for dyes of the fastest types. A brief comparison of the vat dye figures for several years past in terms of per cent shows this trend. The quantity of vat colors produced two years ago, in 1925, for example, was 43 per cent above the 1924 total; that for 1926 was *54 per cent* greater than the output for the year before. Last year American plants turned out more than 5,900,000 pounds of vat colors, a 56 per cent gain over 1925.

The price decline for dyes of all classes, on the other hand, tells a story not so pleasing to our active manufacturers. The average price for 1927 was only 39 cents per pound, lower by 3 cents than that for 1926—and lower by 21 cents than the price for 1922. In five years the average price of American dyes has dropped 36 per cent. Sales of dyes may increase, in other words, and production figures and export figures may soar skyward from year to year, but these signs of increasing business do not indicate that the American dye industry is making more money than five years ago. It is the consumer who is reaping the benefit, in better dyes at lower prices.

TEXTILE STANDARDS FOR A SATISFIED PUBLIC

WHEN homespuns were first bought and sold during the early days of this republic the transactions were extremely simple. The man who wove the fabrics usually dealt directly with the consumers, with those who made them into garments and wore the garments. Here was perfect understanding between the maker and the ultimate purchaser. But the restless spirit that we call Progress gradually made the transactions more complicated. Middlemen set themselves up as wholesalers and retailers, others supplied the weaver with yarns, and still others undertook to color the yarn or dye and finish the fabrics. Goods passed through several hands before the consumer bought them.

Our present textile industry represents the furthest development in this process of making more complex the once simple business of selling fabrics to consumers; and while we may admire the modern efficiency of this great industry, we must admit that a very important quality which the early trade possessed has been lost to us. That quality arose from the almost personal contact of the manufacturer with his consumers. Call the quality goodwill or confidence, or by any other name, its offspring, in any case, was the complete satisfaction of the consumer.

If the selling of textiles to-day were characterized by more of that personal contact and confidence in the manufacturer than we can boast of, there would not exist any of the problems caused by false claims and disputes over qualities like shrinkage, launderability and color fastness.

To express it another way, if the buying public of to-day knew for a fact that when it purchased certain kinds and grades of fabrics it was buying certain kinds and degrees of dye fastness and certain other wearing qualities, that knowledge and a reasonable confidence in trade-marks would forestall any claims, any misunder-

standings, any dissatisfaction; it would reduce such problems almost to nil.

Apparently, even in industry history repeats itself. The need to-day, plainly, is a return to the conditions that governed merchandising during the homespun days—a return to the more intimate confidence between the maker of textiles and his consumers, the public. To use a threadbare simile, the pendulum of merchandising practice has swung all the way and is halfway back. In the arc of its swing lay that near-sighted era in our business life through which we passed at the beginning of this century, when the public was "bedamned"—a condition that can never again exist while the tendency is toward more satisfied consumers.

The textile trade of the future will see the time when the public will be satisfied through more direct contact with the source of its wearing apparel, when consumers, laundries, converters and other groups will have a confidence in graded textiles, founded on an understanding of color fastness and similar qualities.

Practical, hard-headed, dream-scoffing mill men may not be able to see any glimmer of hope for such a Utopia in their industry. They may refuse to believe that any direct contact can ever be set up between the dyehouse of their plant and the dry goods counter of the department store. But those who take that attitude are unreasonable, for an understanding will ultimately be set up with their public and the means will be through that most modern of shibboleths, *standardization*.

The public has taken to standardization in other products and they can be taught to buy their textiles by standards if the manufacturers will set up a system of grading, based on laboratory tests, and abide honestly by that system. That way lies public confidence in fastness claims, confidence that has a dollar-and-cents value higher than mere good-will. Not only can this be achieved but it *will* be achieved eventually. If the present generation of textile men remain so ultra-conservative that they would rather let the problem drop from their shoulders on to those of the next generation, that will be to their misfortune.

The plan of grading fabrics by testing them for fastness and other properties in order that fabrics can be sold by grade is, as most readers of the REPORTER know, one of the principal objects of the Technical Bureau proposed by the president of the American Association of Textile Chemists and Colorists. This may be the very remedy for many, though not all, of the so-called "ills" that prevail in our textile industry to-day. Standardization offers at least a reasonable, definite plan of action to be followed by manufacturers and the merchandising groups; it offers one intelligent answer to the question: How can the mills establish a personal contact with the consumer? On that point alone it deserves a fair and serious trial.

EXTRACTORIALS

All of the huge industries of to-day that have grown in relatively few years from nothing to stupendous size, have based operations of their factories on facts, not opinions. The result has been truly remarkable. The average textile mill in the past has been operated in a rather haphazard, slipshod, careless manner, as compared to the modern industries. We have been governed a great deal by precedent, customs, etc., not always by sound reason; and although we have based our operations and processes mainly on facts, they have also been based to a very large degree on opinions.—*James Cox, before the Textile Division of the A. S. M. E.*

It would be a splendid thing if every textile manufacturer were compelled to own and operate a laundry, a redyeing works and a cleaning plant.—*Canadian Colorist.*

The matter of research invariably makes people think of an anemic young man sitting before a desk pouring over books through horn-rimmed glasses or in some stuffy, out-of-the-way and dark corner musing around with something or other in an atmosphere that certainly wouldn't be inviting to any other form of living thing.

When in reality every business is in its nature a great experimental and research institution. Business has taken a page from the book of science. It has become involved with facts and fact finding, and is no longer a matter of guess and opinion.—*The Starchroom Laundry Journal.*

The business that expects to continue to be maintained at the old stand through several decades and generations will generally be found equipped with suitable research and control laboratories, and to have a definite, established research policy, which is being pursued regardless of transitory business conditions. —*Industrial & Engineering Chemistry.*

I believe that business men are naturally lachrymose. A few are sadistic. They like to get together and talk about bad business in much the same manner that women mobilize and moan about their operations. Half the business ills are imaginary and more than half the operations are unnecessary.—*Edward M. Ruttenber.*

It is instructive to note that, in spite of the remarkable advances in discovery, application and reliability of dyestuffs, not yet has any complete theory of dyeing been universally accepted.—*The Textile Argus.*

The Clavel & Lindenmeyer Plant at Basle*

Learning More About Celanese—Tin Weighting and Other Processes—Laboratories and Research—A Backyard Archeologist on the Rhine

By Chas. E. Mullin, M.Sc., F.C.S., F.A.I.C., F.T.I.
Professor of Textile Chemistry and Dyeing at Clemson College, S. C.

AFTER spending considerable time in Europe visiting plants, universities, textile schools, and other points of interest, I began to think that I had seen about all there was to see of particular interest. Basle, with its numerous dyestuff plants, was of particular interest, but I had heard so much about Dr. Rene Clavel that I wanted to meet him personally. Unfortunately he was in Paris while I was in Basle, but I was so much interested in this scientist and his work that I returned to Basle later especially to meet him. To say that I felt amply repaid is putting it mildly. I was amazed and almost bewildered by the many remarkable things I saw at the Clavel & Lindenmeyer plant.

Of course, you all know a great deal about Celanese. Most of you know that acetate silk has been quite a hobby with me, and I thought I knew most of what there was to know about it, but I must admit that I have a lot to learn. Dr. Rene Clavel, as the patent literature will show to those who doubt, has been a leader in the dyeing and other processing of Celanese for many years. The effects, finishes, colors, designs, etc., that he showed me on Celanese and Celanese combinations were too numerous to describe. And what is more, these were not laboratory samples, but the result of large-scale production in this remarkable plant.

We hear a lot about Celanese in America and some people have expressed doubts regarding its value in the textile industry of the future. All I can say is that I wish some of these doubters had visited the plants of Clavel & Lindenmeyer with me and seen the results they are obtaining on bulk production. Seeing is believing. Personally I have never had any doubts, but now I am a hundred times more enthusiastic about Celanese than I ever was before.

Tin Weighting Celanese

As I said before, I cannot begin to discuss each and every sample shown to me, but Dr. Clavel was kind enough to give me small cuttings from many of these, so that I can show you some of them when I have an opportunity. One thing in particular interested me and that was the tin weighting of Celanese. Clavel & Lindenmeyer have been leaders in the weighting of true silk in Europe for many years, and I have heard it said, but not in their plant, that they have the best tin weighting process for true silk in use anywhere to-day. They weight true silk in every form, from hank to piece goods, and do it with greater accuracy, levelness, and with less operations than I have ever seen or heard of before.

But the weighting of Celanese is another story. I have frequently heard it said that this was impossible, that the use of solvents in such a way was impractical, that the softened and swollen acetate silk could not be handled, etc. Well, as I said before, seeing is believing, and I believe. It not only can be but is being done and on a large scale, at a very rapid rate, too. What is more, Dr. Clavel was not afraid to show me the plant that is being used for this work, as well as the new one just being completed to take care of an even larger production.

Regarding the process itself there is no secret, it is patented and anyone interested can read it in the patent literature, but no one can realize the value of this process until they have seen the product. During the process the Celanese usually gains about 30 per cent, and changes slightly in appearance and handle, as well as quite considerably in its chemical and physical properties. For example, tin weighted Celanese can be dyed at the boil without danger of delustering, with either direct or basic dyestuffs, some of the latter having a surprising and unexpected fastness to light on this fiber. Furthermore, when suitable dyestuffs are used in the dyeing, the tin weighted and dyed Celanese can readily be discharged to a pure white in the usual manner.

Other Effects on Celanese and Other Fibers

But this is only one of the many interesting variations in Celanese handling at the Clavel & Lindenmeyer plant. They have the Simile effects, Joanne d'Arc, artificial leather and a hundred others. Dyeings with colloidal silver and colloidal gold, other special metallic effects, etc. They are simply beyond description and it would have required a special trunk to carry a full line of samples home. However, if you are interested, come to Clemson and see them for yourself.

As before mentioned, this company not only handles Celanese, but also true silk and all other fibers in almost all forms. They are job dyers in the real meaning of the term. They can show you samples of about every variety of fiber combinations from dyed or block printed ribbons, to the finest voiles, four fiber combinations, etc. They not only do this work but manufacture much of their own machinery, have their own art department, engraving department, etc. Of course, this is not all done in one plant. They have several plants in Basle and others scattered through Europe, as well as interests in the United States and Canada.

To say that they have chemical laboratories is to put it mildly. They have them and they also have the brains

*Each instalment of this series is written with the consent of the management of the plant visited, and describes only those processes covered by this permission. No attempt is made to divulge or disclose any secrets or secret processes; however, some of the processes described are undoubtedly covered by patents which may be found in the literature.

in the management to utilize the laboratory results, apply them in the plant, and make the whole thing pay dividends. It would pretty nearly be worth paying the expenses of some textile executives to Europe just to have them see the results of laboratory research in this company's plants.

They not only have chemical control on each and every operation, but Dr. Clavel states that they never attempt any new process without trying it out thoroughly in the laboratory first. It is then investigated on a larger scale in the semi-plant, and finally carried into the plant only when these trials have been successful. As all chemists know, this is the only way to obtain from the laboratory results that pay.

A Fortress on the Rhine

Perhaps some of my friends who have been following the notes on the various plants, schools, etc., are beginning to think that that is about all I have seen in Europe. But just to prove that you are wrong I am going to digress for a few paragraphs to try to describe some interesting points regarding Dr. Clavel's summer home, located on the Rhine about 25 miles from Basle. It is so remarkable in many ways that I believe you will all be interested.

This home is built on the site of an old Roman fortress, located at the extreme northern point of the Roman invasion of, I believe, the first century of the Christian era. How would you like to have the ruins of an old Roman theater in your back yard, with a temple thrown in, too? Well, that's just what I saw, and all located on a high hill overlooking, as any fortress would, the valley of the Rhine.

In excavating at various times in the yard and surrounding grounds, the workmen have unearthed one of the most interesting collections of old Roman articles, ranging from small coins to marble statues and columns, that it has ever been my pleasure to see. And all in Dr. Clavel's own back yard.

I am no artist and if I can't describe the Celanese samples, it is even more beyond me to describe the Italian Renaissance period wood inlay panels and carvings, many of which date back almost to the discovery of America, which are a part of this wonderful home. And to prove that a scientist can also be a business man and a sportsman, as well, I want to add that Dr. Clavel owns and drives the fastest motorboat on the Rhine, as well as an airplane. The motorboat is driven by an airplane engine and propeller. The time spent with Dr. Clavel was alone worth the trip to Europe.

J. Kerns McCallum, of J. & J. McCallum, Ltd., Scotland, arrived on the *Majestic* last week for a short stay in this country. The McCallum company is one of the most prominent dyeing concerns in Great Britain. While in the country Mr. McCallum will visit the plant of the Ontario Dyeing Company, of Philadelphia.

P. SAMUEL RIGNEY

P. Samuel Rigney, secretary of the Roessler & Hasslacher Chemical Company, died October 23 while attending a meeting of the company at Niagara Falls. Mr. Rigney, who was fifty-six, was vice-president of the Special Chemicals Section of the Synthetic Organic Chemical Manufacturers' Association and a member of its board of governors almost from the inception of the association. His sterling qualities and devotion to the best interests of the American chemical industry made him well known among those in the industry who are associated in promoting its welfare.

Services were held 11.30 Saturday morning, October 27, at St. Ursula's Church, Lincoln Avenue, Mt. Vernon, N. Y., followed by interment at the Gate of Heaven Cemetery, Mt. Pleasant, N. Y. Mr. Rigney is survived by his widow, Mrs. Elizabeth V. Rigney, who resides at 11 Willow Place, Mt. Vernon, N. Y.

HINES STILL HEADS COTTON INSTITUTE

Walker D. Hines was re-elected president of the Cotton-Textile Institute at the recent annual meeting of the organization in the Hotel Roosevelt, New York. Robert Armory, of Boston, and Stuart Cramer, of Cramerton, N. C., were also re-elected vice-presidents. Gerrish H. Milliken, of New York City, was re-elected treasurer, and George Sloan, of New York, secretary.

DU PONT AND GRASSELLI CHEMICAL COMBINE

The recent negotiations between E. I. du Pont de Nemours & Co. and the Grasselli Chemical Company, of Cleveland, have resulted in a combination of these two large interests last week. Grasselli is a $56,000,000 corporation, with branches at several points in the East and West. A distributing point in Paterson, N. J., supplies the dyeing trade of that city with textile chemicals used in silk processing.

EXPLOSION IN NEWARK DYE PLANT

A corrugated iron one-story building at the plant of the Dye Products & Chemical Company, 202 Vanderpool Street, Newark, N. J., was demolished on October 17 by an explosion of undetermined origin. The estimated damage was $25,000. The explosion is believed to have been in one of the ovens used in the manufacture of dyes, according to the police. No one was at work at the time.

BUHLMANN BACK FROM EUROPE

Returning last week from an extensive business trip to Europe, A. W. Buhlmann, textile engineer and manufacturer of skein dyeing machinery, was enthusiastic over various new developments in equipment that he saw abroad, as well as over the general business success of his journey.

Technical Notes from Foreign Sources

Dyeing of Acetate Silks

English Patent No. 252,240; *C. M. Barnard and British Alizarine Company, Ltd.*—Covers the dyeing of acetate silks with dyestuffs which contain the radical of acetic acid (not the acetyl radical). Suitable dyestuffs are prepared by the action of diazo-compounds upon phenyl-glycines, or by coupling diazotized amino-phenyl-glycines to ordinary second components. In the anthraquinone series, amino-anthraquinones are converted to glycines by the action upon them of chloracetic or chlorpropionic acid.

Also (English Patent No. 252,646; same authors) dyestuffs are recommended, which contain a glycollic or thioglycollic acid residue, and are prepared by reacting chloracetic acid with phenols or naphthols in presence of alkali Also for the anthraquinone series, chloracetic acid is allowed to react upon the hydroxy- or thio-hydroxy-anthraquinones in the presence of alkali.

Dyeing of Animal Fibers

English Patent No. 267,986; *Durand and Huguenin A.-G.*—Involves the dyeing of animal fibers with the ester salts of the leuco compounds of vat dyestuffs, and the addition of fuller's earth to the oxidizing bath. The fuller's earth may be treated, previous to use, with an acid, to remove any substances which will unite with acids. It is claimed that goods dyed in this way are faster to rubbing.

Increasing of Affinity of Cotton for Acid Dyestuffs

English Patent No. 249,842; *P. Karrer.*—Cotton is partially esterified with arylsulphonic acids (chlorides) such as p-toluene-sulphon-chloride, or similar derivatives of naphthalene, and then treated with ammonia water, aliphatic or aromatic primary, secondary, or tertiary (can this be true?) amines, hydrazines, or hydrazine derivatives. Cotton so treated can be dyed with acid dyestuffs, e. g., Tartrazine. (See also a recent reference to immunized and amidated cotton in these columns. Here partial immunizing is spoken of.)

Bleaching in Combination with Washing

Dr. E. v. Drathen and Dr. K. Sagstetter, Melliand's Textil-Ber. 416, 9 (1928).—The title as given in the original paper reads "Hydrocarbon Preparations in Combination (Mixture) with Sodium Perborate." The authors point out the advantages of combining the processes of washing and bleaching, using a mild bleach, sodium perborate, in mixture with a soap containing benzine, etc.; and from results of their experiments recommend their method strongly.

Oxidation of Cuprammonium Cellulose Solution

L. Kalb and F. von Falkenhausen, Ber. 2514, 60 (1927).—A study of the oxidation of cellulose, dissolved in ammoniacal copper oxide solution, by ammonium persulphate and by potassium permanganate. Oxidation involving less than ½ atom of oxygen to the unit of $C_6H_{10}O_5$ yielded increasing amounts of alkali soluble oxycellulose. With amounts of oxygen over the proportion mentioned, the products obtained were water soluble, and gave the naphthoresorcinol test for glycuronic acid, which was actually isolated in the form of its cinchonine salt from the residue left after dialysis.

The product, when made from filter paper and using 0.03 atom of oxygen, or from cotton using 0.2 atom of oxygen, is entirely soluble in alkali. The copper number rises uniformly with increasing oxidation, but the acidity number rises slowly at first, and afterward more rapidly.

The theories of other investigators are discussed. The authors' conclusions are that in the oxidation of cellulose the alcohol groups are first converted to aldehyde and then to carboxyl groups.

Rotary Power of Cuprammonium Cellulose Solutions

E. Haegglund and F. W. Klingstedt, Annalen 26, 459 (1927).—Hess's statement, that cellulose preparations can be distinguished from each other fairly well by the rotary powers of their complex copper (cuprammonium) compounds are discussed by the authors, and it is brought out that his method does not show the presence of certain impurities. The authors have studied the chemical constitution of cotton, wood and mercerized cellulose, in connection with the rotatory values of their standard preparations.

The optical values of the cotton and mercerized cellulose preparations are in good agreement, but a different value is obtained for wood cellulose. The alkali-resistant part of this, the so-called alpha-cellulose, is believed by the authors to be identical with cotton. Xylose gives rise to a strongly laevo-rotary copper complex. Until more is known about these possibly mixed substances, the optical method of determination must be used with great caution.

From Cotton to Artificial Silk

Dr. A. Koelliker, Zeitsch. f. Farben-Ind. 135, 20 (1928).—A general but clear and useful review of the subject.

DIBRELL, OF MACY'S, GOES TO CELANESE

Edwin R. Dibrell, an executive vice-president of R. H. Macy & Co., Inc., New York, has resigned to become vice-president of the Celanese Corporation of America, a director and a member of its Executive Committee, according to an announcement issued last week by Jesse R. Straus, president of Macy & Co. Mr. Dibrell will assume his new duties some time before the first of 1929. Before coming to Macy's he was a vice-president and senior divisional manager of Lord & Taylor.

RESEARCH FELLOWS

Carolina College Adds Two on Textiles

Arthur H. Thomas, of Fort Mill, S. C., and J. J. Brown, of Weston, Texas, have accepted positions as research fellows in the Textile School of North Carolina State College, Raleigh, N. C.

Mr. Thomas graduated from the Textile School in 1926 and spent some time in the dye laboratory of the Riverside and Dan River Mills, Danville, Va., before going to the Fort Mill Manufacturing Company as a foreman. He will devote his time to dyeing and finishing problems.

Mr. Brown is a graduate of the Textile Department of Texas, A. & M. College, and has had experience in Texas mills. He will devote his time to carding and spinning problems.

Research work is conducted under the direction of Thomas Nelson, Dean of the Textile School. Weaving tests are conducted by the staff of the weaving department.

Louis L. O'Key has joined the selling force of the National Oil Products Company, of Harrison, N. J. He has been assigned to the Southern territory. He was formerly with L. Sonneborn Sons, Inc., of New York City.

The Philadelphia office of American Aniline Products, Inc., has moved from the Bulletin Building, where it has been located for ten years, to 1218 North Front Street. Its new location affords more convenient storage and shipping facilities, with a basement, store and two lofts, offices and a complete laboratory. The entire building has been rented for the present force of this branch.

STATEMENT OF THE OWNERSHIP, MANAGEMENT, CIRCULATION, ETC., REQUIRED BY THE ACT OF CONGRESS OF AUGUST 24, 1912, OF AMERICAN DYESTUFF REPORTER.

Published bi-weekly at New York, N. Y., for October 1, 1928.

State of New York, County of New York, s.s.:

Before me, a notary public in and for the State and county aforesaid, personally appeared Alfred P. Howes, who, having been duly sworn according to law, deposes and says that he is the publisher of the American Dyestuff Reporter, and that the following is, to the best of his knowledge and belief, a true statement of the ownership, management, etc., of the aforesaid publication for the date shown in the above caption, required by the Act of August 24, 1912, embodied in section 411, Postal Laws and Regulations, printed on the reverse of this form, to wit:

1. That the names and addresses of the publisher, editor, managing editor and business manager are:

Publisher—Alfred P. Howes, 90 William Street, New York City. Editor—Louis A. Olney, Lowell, Mass. Managing Editor—Clayton Hoagland, 90 William Street, New York City. Business Manager—Alfred P. Howes, 90 William Street, New York City.

2. That the owner is: Howes Publishing Company, Inc., 90 William Street, New York City; Alfred P. Howes, 90 William Street, New York City; Mary K. Howes, Rutherford, N. J.; J. T. Howes, Rutherford, N. J.; D. C. Howes, Rutherford, N. J.; Cecil R. Howes, Rutherford, N. J.; N. H. Hiller, Carbondale, Pa.; Louis A. Olney, Lowell, Mass.; William F. Collins, Upper Montclair, N. J.; Derfla H. Collins, Upper Montclair, N. J.; William H. Collins, Upper Montclair, N. J.; Joseph L. Schroeder, Hartsdale, N. Y.

3. That the known bondholders, mortgagees and other security holders owning or holding 1 per cent or more of total amount of bonds, mortgages or other securities are: None.

4. That the two paragraphs next above, giving the names of the owners, stockholders and security holders, if any, contain not only the list of stockholders and security holders as they appear upon the books of the company, but also, in cases where the stockholder or security holder appears upon the books of the company as trustee or in any other fiduciary relation, the name of the person or corporation for whom such trustee is acting, is given; also that the said two paragraphs contain statements embracing affiant's full knowledge and belief as to the circumstances and conditions under which stockholders and security holders who do not appear upon the books of the company as trustees, hold stock and securities in a capacity other than that of a bona fide owner; and this affiant has no reason to believe that any other person, association or corporation has any interest, direct or indirect, in the said stock, bonds or other securities than as so stated by him.

Alfred P. Howes, Publisher.

Sworn to and subscribed before me this 20th day of September, 1928.

[Seal] Caroline Essenbreis.

Notary Public, Kings County, N. Y.

(My commission expires March 30, 1929.)

The rate for "Position Wanted" advertisements in this column is 2 cents a word—with a minimum of 50 cents per insertion. For all other types of advertisements—i. e., help wanted, machinery or supplies for sale—the rate is $5.00 per column inch or less per insertion.

PIECE GOODS DYERS' SALES EXECUTIVE

Will secure new business, silk weighting and dyeing, rayons, etc. Large following New York mill agents and converters. Commission. Address: Classified Box 484, American Dyestuff Reporter.

LABORATORY ASSISTANT

Wanted by old established dyestuff manufacturer young man, assistant in application laboratory. Must have experience in testing of dyestuffs on textiles, paper and leather, also knowledge of printing. State age, experience and salary. Address: Classified Box 487, American Dyestuff Reporter.

SALESMAN

Position open by a leading dyestuff concern for a young man with experience in selling aniline colors in New England territory. Must have ambition to hustle for business. Reply with full particulars as to age, ability and salary expected. Address: Classified Box 488, American Dyestuff Reporter.